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Contents

Abstract

The production of farmed fish has grown continuously, and consumption of farmed fish has become substantial as compared to wild fish. Well-known species are salmon, trout and shrimps, whereas recently new species like tilapia and pangasius were introduced. There is only limited information on the contamination of farmed fish with pollutants. The information mainly focuses on the well-known species (e.g. salmon, trout and shrimp) and the well-known pollutants (e.g. polychlorinated dibenzo-*p*-dioxins and –furans (PCDD/Fs), polychlorinated biphenyls (PCBs) and heavy metals). Nearly no information is available on the contamination of new species (e.g. pangasius and tilapia) and emerging contaminants like polybrominated diphenyl ethers (PBDEs), hexabromocyclododecane (HBCD), perfluorinated compounds (PFCs). Furthermore, there is limited information available on the contamination of fish feeds by toxaphene and mycotoxins.

This study was initiated to investigate a wide range of pollutants in the top five consumed fish in the Netherlands (salmon, trout, tilapia, pangasius and shrimps). Farmed fish samples were collected from different sources (supermarkets, fish stores, markets and suppliers for restaurants) and analysed for PCDD/Fs, PCBs, organochlorine pesticides (OCPs), PBDEs, HBCD, PFCs, heavy metals and residues of antibiotics. Furthermore, fish feeds and ingredients were collected and investigated for mycotoxins and toxaphene residues.

This extensive study shows that contaminant levels in the Dutch consumption top-5 farmed fish (salmon, trout, tilapia, pangasius and shrimps) are very low (mostly < 1 ng/g wet weight), and far below the applicable legislative limits. The contaminant levels decrease in the following order PCBs ≈ OCPs > PBDEs ≈ HBCD ≈ PFCs > PCDD/Fs and dioxin-like (dl)-PCBs. Generally, the contaminant levels decrease like: salmon > trout > tilapia ≈ pangasius ≈ shrimp. Levels in farmed fish are generally lower than levels observed in wild fish. The levels of heavy metals were also well below the applicable legislative limits.

Toxaphene levels (sum of chlorobornanes (CHBs) 26, 50 and 62) in feeds and feed ingredients are well below the legislative limits. Frequent monitoring of feeds is therefore not of high priority. Of the mycotoxins, fumonisins, zearalenon (ZEN) and deoxynivalenol (DON) were frequently detected in feed ingredients and feeds. It is not clear if mycotoxins accumulate from the feeds into the edible fish tissues. More research is needed to investigate this.

The Dutch consumption of farmed fish is increasing, and within this group, the share of pangasius and tilapia is growing rapidly. Considering the low contaminant levels observed in pangasius and tilapia, it is believed that the human exposure to PCBs, OCPs, PBDEs, HBCD, PFCs and dioxins and dl-PCBs through fish consumption will decrease.

1. Introduction

During the last decades the world production of aquaculture has grown considerably. In relation with that, the human consumption of farmed fish and crustaceans is also increasing, on one hand of well-known species such as salmon and shrimp and on the other hand of new species like pangasius, tilapia, sole and cod (van Diemen and van Dongen 2008). Because comprehensive information on farmed fish was lacking, the Dutch Food and Consumer Product Safety Authority (VWA) and the Ministry of Agriculture, Nature management and Food Quality (Min. LNV) initiated three studies on farmed fish:

1. Trade flows of farmed fish in the Netherlands;
2. Antibiotic resistance of farmed fish;
3. Chemical contaminants in farmed fish and their feeds.

The focus in these studies was on farmed fish species and shrimps that are fed artificial feed. Bleu mussels (*Mytilus edilus)* are sometimes also regarded as aquaculture products, but they were not included because they grow on nutrients from the surrounding waters rather than on artificial feed. The same holds for farmed tuna, which is fed with fish and not with artificial feeds. In this report, the results of the study “Chemical contaminants in farmed fish and their feeds” are presented.

Recent reports have shown that farmed salmon, trout and shrimp can be contaminated with a range of contaminants including polychlorinated dibenzo-p-dioxins and –furans (PCDD/Fs), polychlorinated biphenyls (PCBs), organochlorine pesticides (OCPs) and polybrominated diphenyl ethers (PBDEs), hexabromocyclododecane (HBCD), heavy metals and residues of antibiotics (Zennegg et al. 2003;Hites et al. 2004;Hites et al. 2004;Hayward et al. 2007;Hastein et al. 2006; Fauconneau 2001). On the contamination of recently introduced species like tilapia and pangasius, virtually no contaminant information was available. Fish feed and fish feed ingredients can be contaminated as well. The contamination of livestock feeds and feed ingredients with mycotoxins is commonly observed (Yiannikouris and Jouany 2002), and it was assumed that this would also hold for aquaculture feeds (although no comprehensive information was available).

Three major knowledge gaps can be identified: (1) Few reports exist on the contamination of farmed fish with PBDEs and none on perfluorinated compounds (PFCs) and HBCD, (2) Virtually no information is available on contamination of tilapia and pangasius with heavy metals, residues of antibiotics, PCBs, PCDD/Fs, OCPs, PBDEs, HBCD and PFCs, and (3) Virtually no information is available on mycotoxin contamination of aquaculture feeds. This study aims to fill these gaps by producing a comprehensive dataset.

2. Materials and methods

2.1 Sampling and sample pre-treatment

The fish species selected for sampling were based on information from the project 'Trade flows of farmed fish", performed by Innotact Consulting BV (van Diemen and van Dongen, 2008). From this report, it is clear that the current top-5 farmed species consumed in The Netherlands are salmon, trout, shrimp, tilapia and pangasius. Pangasius is becoming increasingly popular and it is expected that the consumption will increase in the next years. Furthermore, a sample of farmed cod was included as well. In monitoring programs, cod contains low levels of contaminants and as such can be regarded as a 'reference' fish. It was assumed that farmed cod could also act as a 'reference' sample. Details on the consumption volumes (van Diemen and van Dongen, 2008), contaminants and number of samples can be found in Table 2.1. Dioxins were not investigated in salmon and trout because literature data (van Leeuwen et al., 2008a) showed that the levels commonly observed (world-wide) are below the EU MRL (EC, 2006a).

Table 2.1. Sample overview on farmed fish and shrimp

				Number of samples investigated							
Species	Latin name	Consumption (tons/yr)*	Origin	PFCs	PBDEs	HBCDs	PCDD/Fs and dl-PCBs	PCBs	OCPs	Heavy metals	Antibiotics
Salmon	Salmo salar	8700	Norway, Scotland, Chili	7	7	7	-	7	7	-	6
Pangasius**	Pangasius hypophthalmus	1700	Vietnam	7	7	7	5	7	7	7	4
Tilapia	Oreochromis mossambicus, Oreochromis niloticus	1200	China, Ecuador, Indonesia, Netherlands	7	7	6	5	7	7	7	4
Trout	Oncorhynchus mykiss, Salmo trutta	900	Denmark, Italy, Turkey	5	5	5	-	5	5	5	5
Shrimps	Penaeus monoden, Litopenaeus vannamei	1500	Bangladesh, Mixed-Asia***, Netherlands	6	6	6	5	6	6	-	5
Cod	Gadus Morhua	N.a.	Norway	1	1	1	1	1	1	1	1
	Total	14000		33	33	32	16	33	33	20	25

N.a.: Not available

* Dutch consumption of farmed fish in 2006 (ton/yr), representing approx. 18% of the total fish and shellfish consumption in the Netherlands (van Diemen and van Dongen, 2008). No consumption figures for farmed cod were available.

** Other names commonly used world-wide are Swai, Sutchi catfish, Striped catfish and Iridescent shark.

*** Mixed origins were declared on the package label (Bangladesh/India, Indonesia/China or Thailand/Malaysia/ Indonesia).

Detailed sample information, including Laboratory Information Management System (LIMS) identities, sample weights, physical state at purchase etc. can be found in Appendix 1. The fish and shrimp samples were bought between October 2007 and January 2008 from various suppliers from different places in The Netherlands. These included supermarkets, fish stores, week markets and suppliers for restaurants. One shrimp sample (07/871) was obtained directly from the farm. Pangasius and tilapia samples were purchased as whole fillets, salmon and cod were purchased as parts of the whole fillets and trout was purchased as whole fish but with intestines being removed. These trout samples were filleted. One trout sample (07/869) was bought as fillets. All fillets were pooled per sample. Shrimps were purchased in a variety of physical states. Some samples were cooked and shell was completely removed (except for the tail, 07/789 and 07/811), whereas others were not cooked and with skin and heads still on (07/797 and 07/871). In the latter cases, the heads were removed (but skin left on) prior to pooling the individuals. All samples were stored at –20°C in their original packaging. After thawing, small subsamples were taken for the heavy metal analysis by cutting a part of each fillet using a titanium knive on a acrylic glass board (poly(methyl methacrylate)). All subsamples were stored in a polypropylene container that was cleaned by acid and ultrapure water. After subsampling for heavy metals, the remaining material was ground and homogenised using a kitchen machine (Type AL2-3, Krefft Gmbh, Gevelsberg, Germany) equipped with a rotary knife and sieve with 10 mm diameter holes. The homogenised samples were stored in glass containers at –20°C until analysis. Subsamples were sent to RIKILT for analysis of dioxins and dl-PCBs and subsamples were sent to the VWA laboratory for analysis of residues of antibiotics.

The feed and feed ingredients sampling scheme for investigating toxaphene and mycotoxins was developed in consultation with representatives of Nutreco and of the VWA. Feed and feed ingredient samples were taken by the feed sampling team of the VWA, of which subsamples were sent to the IVM laboratory (for toxaphene analysis). Additional ingredients were obtained from Peterson/TLR (Rotterdam), which were sent to the IVM laboratory for toxaphene analysis (and subsamples were sent to the VWA laboratory for analysis of mycotoxins). The sample overview is shown in Table 2.2 for feed ingredients and in Table 2.3 for compound feed.

Table 2.2. Feed ingredient samples.

IVM LIMS	VWA sample code	Sample type	Toxaphene	Mycotoxins
08/0395	N.a.	Wheat (UK)	N.a.	Yes
08/0396	N.a.	Soy bean (US/Canada)	N.a.	Yes
08/0397	N.a.	Corn (Brasil)	N.a.	Yes
08/0398	N.a.	Pea meal (EU)	N.a.	Yes
08/0399	N.a.	Rapeseed (NL)	N.a.	Yes
08/0400	N.a.	Lupines (Australia)	N.a.	Yes
08/0401	N.a.	Fish meal	N.a.	Yes
08/0402	N.a.	Palm oil (Indonesia)	N.a.	N.a.
08/0403	N.a.	Sunflower oil (Ukraine)	Yes	N.a.

08/0404	N.a.	Lineseed oil	Yes	N.a.
08/0405	N.a.	Rapeseed oil	Yes	N.a.
08/0406	N.a.	Soy oil	N.a.	N.a.
08/0407	N.a.	Fish oil	N.a.	N.a.
08/0408	N.a.	Soy bean meal (Argentina)	N.a.	Yes
08/0356	66251969	Fish meal (Norway)	Yes	Yes
08/0357	66251977	Fish meal (Peru)	Yes	Yes
08/0364	66251993	Palm oil	Yes	Yes
08/0365	66251985	Fish oil	Yes	Yes
		Total	7	13

N.a.: Not applicable

Table 2.3. Compound feed samples.

IVM LIMS	VWA sample code	Sample type	Toxaphene	Mycotoxins
08/0358	66252035	Catfish (1)	Yes	Yes
08/0359	66252043	Catfish (2)	Yes	Yes
08/0371	66252159	Catfish (3)	Yes	Yes
08/0355	66251497	Catfish / Tilapia	Yes	Yes
08/0362	66252027	Tilapia (1)	Yes	Yes
08/0363	66252019	Tilapia (2)	Yes	Yes
08/0370	66252132	Tilapia (3)	Yes	Yes
08/0360	66252051	Trout (1)	Yes	Yes
08/0361	66252078	Trout (2)	Yes	Yes
08/0367	66252108	Trout (3)	Yes	Yes
08/0366	66252094	Salmon	Yes	Yes
08/0368	66252116	Common carp	Yes	Yes
08/0369	66252124	Ide	Yes	Yes
08/0372	66252167	Eel	Yes	Yes
08/0373	N.a.	Shrimp	Yes	Yes
		Total	15	15

2.2 Chemical analysis

A wide range of contaminants were analysed in the fish and feed samples. These are mentioned in Table 2.4 (farmed fish and shrimps) and Table 2.5 (fish compound feed and feed ingredients).

Table 2.4. Contaminants analysed in farmed fish and shrimps

Compound class	Individual compounds
polychlorinated dibenzo-*p*-dioxins and –furans (PCDD/Fs) and dioxin-like polychlorinated biphenyls (dl-PCBs)	All 29 WHO PCDD/F and dl-PCB congeners (van den Berg et al., 2006)
Polychlorinated biphenyls (PCBs)	Congeners 28, 52, 101, 118, 138, 153 and 180
Organochlorine pesticides (OCPs)	Hexachlorobutadiene (HCBD), pentachlorobenzene (QCB), hexachlorobenzene (HCB), α-hexachlorocyclohexane (α-HCH), β-HCH, γ-HCH, heptachlor, trans-heptachlor epoxide, cis-heptachlor epoxide, aldrin, telodrin, isodrin, dieldrin, endrin, α-endosulfan, o,p'-DDE, p,p'-DDE, o,p'-DDD, p,p'-DDD, o,p'-DDT and p,p'-DDT
Polybrominated diphenyl ethers (PBDEs)	Congeners 28, 49, 71, 47, 66, 77, 100, 119, 99, 85, 126, 154+bromobiphenyl (BB)153, 153, 138, 156, 184, 183, 191, 197, 196, 208, 206, 209
Hexabromocyclododecane (HBCD)	α-, β- and γ-diastereomers
Perfluorinated compounds (PFCs)	Perfluorohexanoic acid (PFHxA), perfluoroheptanoic acid (PFHpA), perfluorooctanoic acid (PFOA), perfluorononanoic acid (PFNA), perfluorodecanoic acid (PFDA), perfluoroundecanoic acid (PFUnA), perfluorododecanoic acid (PFDoA), perfluorobutane sulfonate (PFBS), perfluorohexane sulfonate (PFHxS), perfluorooctane sulfonate (PFOS) and perfluorinated sulfonamide (PFOSA)
Heavy metals	Mercury (Hg), lead (Pb) and cadmium (Cd)
Residues of antibiotics (I)	Tetracyclines, (fluoro-)quinolonen, macroliden en beta-lactams, Nitrofuranes, Chloramphenicol
Residues of antibiotics (II)	*Sulfonamides*: Sulfadoxine, Sulfamonomethoxine, sulfamoxole, Sulfamethoxazol, Sulfadiazine, Sulfadimidine, Sulfadimethoxine, Sulfaquinoxaline, Sulfachloorpyridazine, Sulfamethizole, Sulfamethoxypyridazine, Sulfapyridine, Sulfasoxazole, Sulfanilamide Sulfisomidine, sulfametomidine, Sulfametoxydiazine, Sulfaperine, Sulphenazole, Sulfamylon (mafenide), Prontosil, Sulfacetamide, Sulfasalazine, Phtalylsulfathiazole, Succinylsulfathiazole, Sulfalene, Sulfathiourea, Sulfaguanidine, Sulfamerazine, Sulfathiazole. Trimethoprim

Table 2.5. Contaminants analysed in fish compound feed and feed ingredients

Toxaphene	Chlorobornanes (CHB) 26, 50 and 62
Mycotoxins	Aflatoxin B1, Aflatoxin B2, Aflatoxin G1, Aflatoxin G2, Ochratoxin A, Deoxynivalenol (DON), Fumonisin B1, Fumonisin B2, Fumonisin B3, Nivalenol, Diacetoxyscirpenol (DAS), T2-Toxin, HT2-Toxin, 3-Ac-Don, Zearalenone (ZEN), 15-Ac-Don, Penicillic acid, Fusarenon X, Ergotamin, Roquefortin C, ß-Zearalanol (ß-ZAL), α-Zearalanol (α-ZAL), Citrinin, Zearalanone (ZAN), Cyclopiazonic acid, Sterigmatocystin, α-Zearalenol (α-ZEL), Mycophenolic acid, Alternariol, Alternariol methyl ether, Ergonovin, Ergocornin, α-Ergocryptin, Ergocristin

Lipid determination – The lipid determination was performed according to a modified Bligh and Dyer method (de Boer 1988). This method determines both the triglycerides as well as the more polar lipid compounds such as phospholipids and sterols, and is therefore suitable for both lean and lipid-rich fish.

*Dioxins and dl-PCBs – A*ll samples were extracted using an accelerated solvent extraction system (ASE 200 Dionex). Prior to extraction sixteen ^{13}C labelled dioxins, four ^{13}C labelled non-ortho PCBs, eight ^{13}C labelled mono-ortho PCBs internal standard were added to the samples. Biological samples, including fish, were extracted three times with hexane/acetone (7:3, v/v) at 100°C and 1500 PSI during 10 minutes. Extracts were concentrated down to < 0.1 ml and after addition of the ^{37}Cl-2,3,7,8-TCDD (clean up standard) extracts were made up with hexane to 25 ml. Extracts were purified by a comprehensive automated system, the so called Power-Prep™ (Fluid Management Systems, Waltham, USA). Extracts were transferred to the Power-Prep system and purified on an acid silica column, a neutral silica column, a basic alumina column and an activated carbon/celite column. Custom made solvents and mixtures were used for elution; hexane, hexane/dichloromethane (1:1, v/v) ethylacetate/toluene (1:1, v/v) and toluene (the program can be downloaded from the RIKILT website). The volume of the final extract was reduced to 0.5 ml using a turbovap. The recovery standards ^{13}C 1,2,3,4-TCDD and ^{13}C 2,3,4,6,7,8-HxCDF were added and the volume of the extract was again reduced to 0.5 ml using a turbovap. PCDD/Fs and PCBs analyses were performed by GC-HRMS using an Agilent (Wilmington, USA) 6890 Series gas chromatograph and an AutoSpec Ultima high resolution mass spectrometer (HRMS) (Waters, Milford, USA). The GC column was a DB5 MS (60 m, 0.25 mm i.d., 0.25 μm; J&W, Folson, USA). The mass spectrometer was operated in the electron impact ionization mode (EI), using selected-ion monitoring (SIM). The mono-ortho and indicator PCB fraction was injected (2 μl, splitless). The PCDD/F's and non-ortho-PCB containing fraction (100 μl) was injected by a CIS-3 PTV injector in the solvent-vent mode with a vent flow of 100 ml/min and a vent pressure of 100 Pa. The initial temperature of the PTV was 70 °C. After injection the temperature of the PTV was raised to 280 °C with 720 °C/min.

PCBs and OCPs – The PCBs and OCPs (mentioned in Table 2.4) were Soxhlet-extracted (dichloromethane (DCM)-acetone 3:1 v/v, 16 h) from the sample. Internal standard CB 103 was added after extraction. The co-extracted lipids were removed by Al_2O_3-column chromatography (15 g, 8% w/w H_2O, eluted with 170 ml n-pentane) and subsequently fractionated over a silica column (1.8 g, 1.5% w/w H_2O, PCB fraction eluted with 14 ml n-hexane and the OCP fraction eluted with 10 ml n-hexane-diethylether (DEE) 85:15

v/v). After a final concentration step (to approx. 500 µl), both fractions were analysed on a dual column GC-electron capture detection (ECD) system. 1 µl of the extract was injected in a split-splitless injector operated in the pulsed splitless mode (injector operated at 250°C). A pressure pulse (280 kPa, 1.5 min) was used for rapid transfer of the analytes to the columns. The columns used were CP-Sil-8 CB (50 m x 0.2 mm id x 0.33 µm film) and CP-Sil-19 CB (custom made, 50 m x 0.2 mm id x 0.33 µm film). They were both inserted in the injector using a 2-hole ferrule. The column flow was 1 ml/min (helium). Because the OCP fraction of the pangasius and tilapia samples contained interferences, they were treated with concentrated sulphuric acid (H_2SO_4) and re-analysed. In these cases, the drins were quantified from the untreated fraction, whereas the other OCPs were quantified in the treated fraction. CB 103 was added as internal standard after extraction to correct for the clean up and GC analysis procedure.

PBDEs and HBCDs - PBDEs and HBCDs were Soxhlet-extracted (16 hrs) from the matrix using a 3:1 dichloromethane (DCM):acetone (v/v) mixture. After extraction, the following IS were spiked to the sample extract: $^{13}C_{12}$-α-, β- and γ-HBCD, $^{13}C_{12}$-deca-BDE and BDE 58. The lipids were removed from the crude extract by acid-silica column chromatography (20 g, 40% w/w H_2SO_4, elution with 150 ml DCM:n-hexane 3:7 v/v). The eluate was fractionated over a silica column (1.8 g, 1.5% w/w H_2O, 1st fraction eluted with 14 ml n-hexane and subsequently with 25 ml n-hexane-diethylether 85:15 v/v for the PBDEs and a part of HBCD, and with 10 ml DEE (2nd fraction) for the rest of the HBCD. After concentration of the purified extract to 500 µl, the BDEs were analysed by GC-ECNI-MS (Agilent 6890, Wilmington, USA). The column used was CP-Sil-8 CB (50 m x 0.25 mm id x 0.25 µm film). Deca-BDE was analysed on a short column (DB-5, J&W, Folson, USA, 15 m x 0.25 mm id x 0.25 µm film) in order to prevent degradation due to long residence times in the GC oven at high temperatures (de Boer and Wells, 2006). The BDEs were detected using the bromine isotope (m/z 79 and 81) except for deca-BDE which was quantified based on the molecular ion (m/z 486, and m/z 494 for the ^{13}C internal standard). After analysis of the BDEs by GC, fraction 1 was combined again with fraction 2 (containing the rest of HBCD), carefully evaporated to dryness and redissolved in 100 µl acetonitrile:water 75:25 (v/v). The HBCD diastereomers were analysed by HPLC-ESI-MS/MS using a Zorbax eclipse 2.1 x 30 mm, 3.5 µm particles analytical column and a Zorbax eclipse 2.1 x 12.5 mm, 5 µm particles (both from Agilent, Wilmington, USA). The diastereomers were quantified using MRMs.

PFCs –The targeted compounds of this study are listed in Table 2.4. Prior to extraction, $^{13}C_n$-analogues of PFHxA, PFNA, PFDA, PFUnA and PFOS were added (all Wellington Laboratories, Guelph, Ontario, Canada) as well as $^{18}O_2$-PFOSA (RTI International, NC, USA). Prior to extraction, the samples were dried by mixing with Kieselguhr. Extraction was performed with 10 ml methanol and shaking for 30 minutes. This was repeated once with 5 ml fresh methanol (MeOH). After combining the extracts and reducing the volume under N_2 stream, a clean-up was performed according to the method first published by Powley et al. (Powley et al., 2005; Powley and Buck, 2005). The PFCs were chromatographically separated on a Symmetry C18 (50 x2.1 mm, 5 µm particle size, kept at 20°C), which was preceded by a Symmetry C18 (20 x 3.9 mm, 5 µm particle size) (Waters, USA). The eluent consisted of (A) 2 mM ammonium acetate in water and (B) methanol. After dilution of the methanol extract 1:1 with ultrapure water, the extracts were analysed on an Agilent 1200 HPLC coupled with an Agilent 6410 ESI-MS/MS system. The system was equipped with a degasser and an autosampler. The injection volume was 20 µL. The capillary voltage was set at 1000 V, the nebuliser at 25 PSI, the gas flow at 6 L/min and the gas temperature was set at 325°C. The samples were quantified using MRMs.

Heavy metals – The fish and shrimp samples are lyophilised in acid-prewashed plastic cups. The resulting powder is homogenised and 0.5 g of material was digested with 4 ml 70.5% nitric acid and 12 ml 36.5-38% hydrochloric acid, using a CEM MDS 2000 microwave (Matthews, NC, USA). After the first digestion, 1 ml of hydrogen peroxide was added and a second digestion was performed. The digested samples were sent to Omegam Laboratories, Amsterdam for analysis by inductive coupled plasma (ICP)-MS.

Toxaphene – The feed and feed ingredient samples were Soxhlet extracted using n-pentane-DCM (1:1 v/v). The co-extracted lipids (500 mg) were removed by Al_2O_3-column chromatography (25 g, 8% w/w H_2O, eluted with 250 ml n-pentane) and subsequently fractionated over a silica column (2.5 g, 2% w/w H_2O). The 1st fraction (14 ml iso-octane) is discarded, and the 2nd fraction (12 ml n-hexane-DEE 80:20 v/v) is collected and treated with concentrated H_2SO_4 for removal of interferences. The concentrated extracts were injected on the same GC-ECNI-MS instrument as used for the PBDEs, and equipped with a CP-Sil-8 CB column (50 m x 0.25 mm id x 0.25 µm). Quantification was performed on m/z 377 (CHB 26), 413 (CHB 50) and 377 (CHB 62).

Mycotoxins – The mycotoxins were analysed according to a multimethod recently published by Spanjer et al. (Spanjer et al., 2008). Samples were dry milled. Of the ground sample material, 25 g was mixed with 100 ml acetonitrile/water (80:20 v/v) and placed in the horizontal shaker for 2 h. One ml of the clear extract is diluted with 3 ml water and mixed. Cloudy solutions were filtrated using a 0.45-mm membrane filter (Schleicher & Schull, Spartan 13). Final extracts were injected on the LC-ESI-MS/MS system. The LC–ESI-MS/MS system consisted of a Waters Alliance 2695 separation module with a 100-ml injection loop (Waters, Milford, MA, USA) coupled to a Quattro Ultima triple quadrupole mass spectrometer (Waters-Micromass, Manchester, UK) equipped with an electrospray interface. A 20-ml aliquot was injected on an Alltima C18 (150x 3.2 mm, 5 mm) column (Alltech, Breda, The Netherlands) at 30°C column temperature. The gradient was composed of solvents A (0.1% formic acid in water) and B (0.1% formic acid in acetonitrile) at a flow-rate of 0.3 ml/min. Further details can be found elsewhere (Spanjer et al., 2008).

Residues of antibiotics – The presence of ABs (sulfonamides, tetracyclines, quinolones, macrolides and beta-lactams) was initially investigated by a microbiological screening approach. The liquid fraction of each sample was applied to a medium with several micro-organisms that respond to the different ABs present in a sample. This method is able to detect the presence of the above-mentioned ABs at a level below the EC-MRL.

Some samples showed a positive response for sulfonamides, beta-lactams and macrolides, and required confirmation by instrumental methods. Confirmation of the presence of macrolides (erythromycine, josamycine, lincomycine, spiramycine, tylosine, oleandomycine, pirlimycine, tiamulin, tilmicosine, valnemulin en tulathromycine) was performed by LC-ESI-MS. Confirmation of the presence of beta-lactams was performed by treatment of the sample with penase, combined with the microbiological assay. Confirmation of the presence of sulfamethoxazole, sulfamethazine,c sulfadiazine and sulfapyridine was performed by HPLC and UV detection. In addition, a wider group of sulfonamides (31 compounds, see Table 2.4) was screened for using UPLC-ESI-ToFMS.

Chloroamphenicol was screened for using an ELISA assay, which is suitable for screening samples at the MRL level. Eleven suspect samples were analysed by LC-MS for confirmation.

The nitrofurans were screened for using an LC-MS/MS method. The nitrofurans were released from the tissue by acid hydrolysis and derivatisation to the nitrophenyl derivative (using nitrobenzaldehyde). The compounds analysed were AOZ (amino-oxazolidone), AMOZ (amino morpholino oxazolidone), AHD (amino hydantoine) and SEM (semicarbazide). The method is capable of screening at a level just below the EU-MRL of 1 µg/kg.

Quality Assurance

The quality of the analysis was assured routinely by analysis of procedural blanks, duplicate analysis of selected samples, internal reference materials, certified reference materials (CRMs) (mussel tissue standard reference material (SRM) 2978 for PCBs and OCPs and the candidate CRM BROC-01 for the PBDEs, DOLT-2 (shark liver, Environment Canada) for heavy metals), the use of (mass labelled) internal standards (as mentioned above), recovery experiments by spiked samples (toxaphene, antibiotics) and the participation in various interlaboratory studies (e.g. Folkehelsa (www.fhi.no), QUASIMEME (www.quasimeme.org), FAPAS and the 2nd world-wide PFC interlaboratory study (van Leeuwen et al., 2008b) with satisfactory results.

3. Results and discussion

3.1 Contaminant concentrations in fish

3.1.1 Dioxins and dl-PCBs

The levels detected in pangasius, tilapia and shrimps were very low (Figure 3.1, top). On an upperbound basis, the levels were approximately 0.2 pg total-TEQ/g ww (the Dutch shrimp sample was slightly higher). These levels are well below the EU-MRL of 8 pg total-TEQ/g ww (EC, 2006a). The lowerbound concentrations (Figure 3.1, bottom) ranged from <1 to 82 fg total-TEQ/g ww, and nearly all congeners were below the LOQ (except for e.g. CB 77).

Salmon and trout were not analysed for PCDD/Fs and dl-PCBs as in a literature survey (van Leeuwen et al., 2008a) it was shown that a substantial amount of data was available showing that these fish met the EU MRL of 8 pg total-TEQ/g ww. As compared to other fish, the levels are much lower than those observed in an earlier study on dioxins and dl-PCBs in Dutch wild marine and freshwater fish (van Leeuwen et al., 2007). In 2007, Hoogenboom et al. reported results for wild eel with different sizes from various locations in The Netherlands (Hoogenboom et al., 2007). The levels found there were high (up to 75 pg total-TEQ/g ww), and confirmed earlier findings (van Leeuwen et al., 2007).

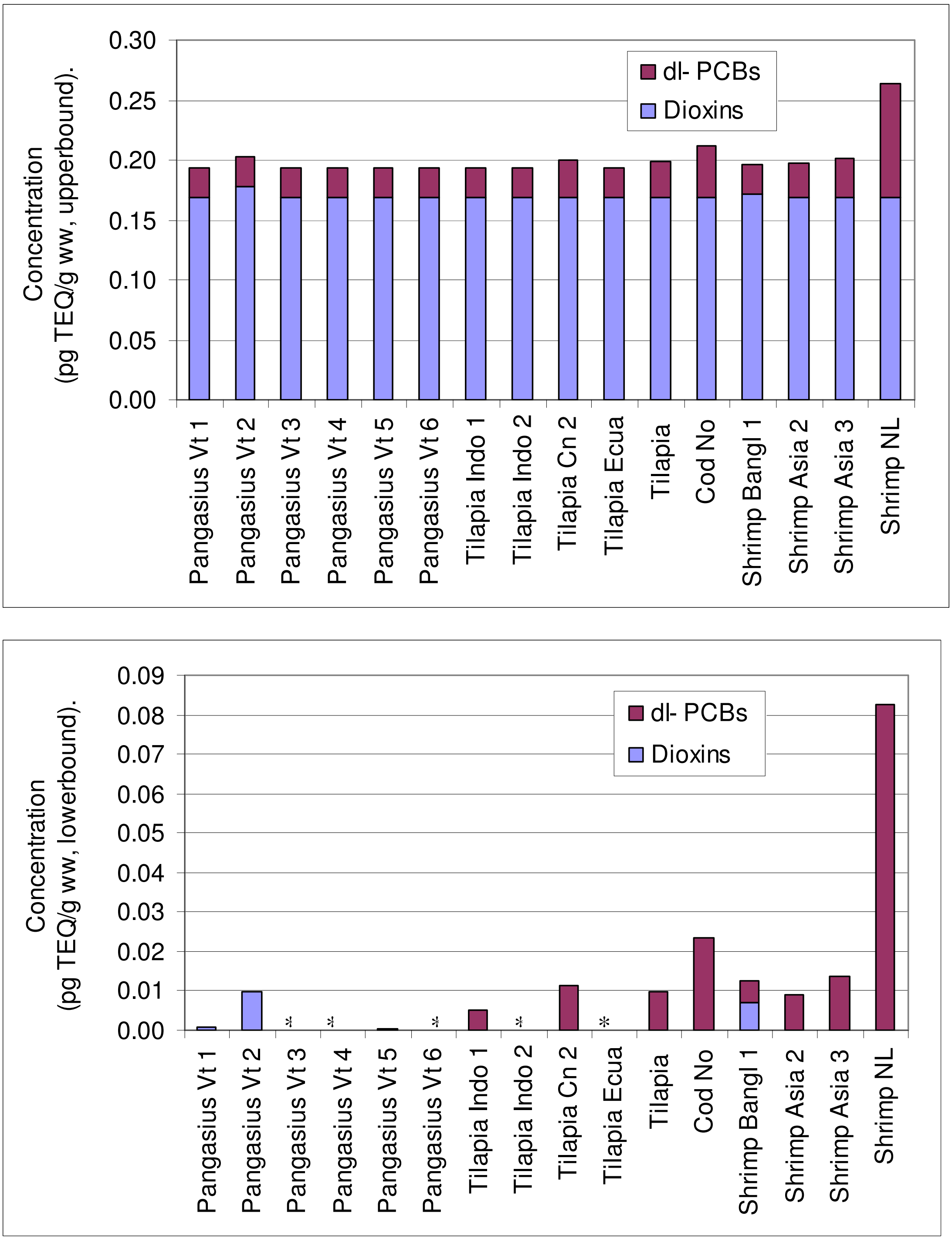


Figure 3.1. *Dioxins and dl-PCBs in pangasius, tilapia, cod and shrimp. In the top graph, upperbound data is presented, whereas lowerbound data is presented in the lower graph. Country codes: No = Norway, Asia = Asia mixed origins, Bangl = Bangladesh, NL = Netherlands, Vt = Vietnam, Ecua = Ecuador, Cn = China and Indo = Indonesia. Samples indicated with * may appear below LOQ, but did contain detectable (but low) amounts for some congeners (e.g. CB 77). Individual sample results are shown in Appendix 2.*

3.1.2 PCBs and OCPs

Data on PCBs and a selection of OCPs is shown in Figure 3.2. The PCB levels in all fish samples met the MRLs for the seven indicator PCBs as laid down in the Dutch Food Law (‘Warenwet’) (Anon. 1984). For OCPs, no MRLs are available, but generally, the sum-DDT levels were in the same range as the sum 7 PCB levels. Dieldrin and HCB concentrations were lower. The levels in the carnivorous fish (salmon, trout) were higher than those of the omnivorous pangasius and tilapia and shrimp. Apart from the OCPs in Figure 3.2, α-HCH was detected in some tilapia and pangasius samples, α-endosulfan was found in the salmon sample from Chile and QCB and HCB were detected in several samples. Several other OCPs were not detected in any of the samples (β-HCH, γ-HCH, HCBD, heptachlor epoxide, aldrin, telodrin, isodrin, o,p’-DDE, cis-HEPO and trans-HEPO). Compared to the study by Hites et al. (Hites et al., 2004a), the OCP levels in salmon are at the lower end of the range they reported (see Figure 3.1). This suggests that presumably the aquaculture industry nowadays uses less contaminated feeds and the salmon diet may nowadays consist of a lower proportion of fish meal and fish oil. In accordance with the Hites study, PCB and OCP levels in salmon from Chile were lower than those from Europe. The PCB levels in the Hites study ranged from 10-60 ng/g for the sum of 197 congeners (Hites et al., 2004a). The PCB levels in aquaculture samples from Belgium (mainly trout) were 50-60 ng/g, which is much higher than those observed in this study, whereas the DDT levels are in the same range. The PCB levels in our study are much lower than in those studies.

The PCB and OCP levels are much lower than those observed in Dutch wild freshwater and marine fish (van Leeuwen et al., 2007; van Leeuwen et al., 2006).

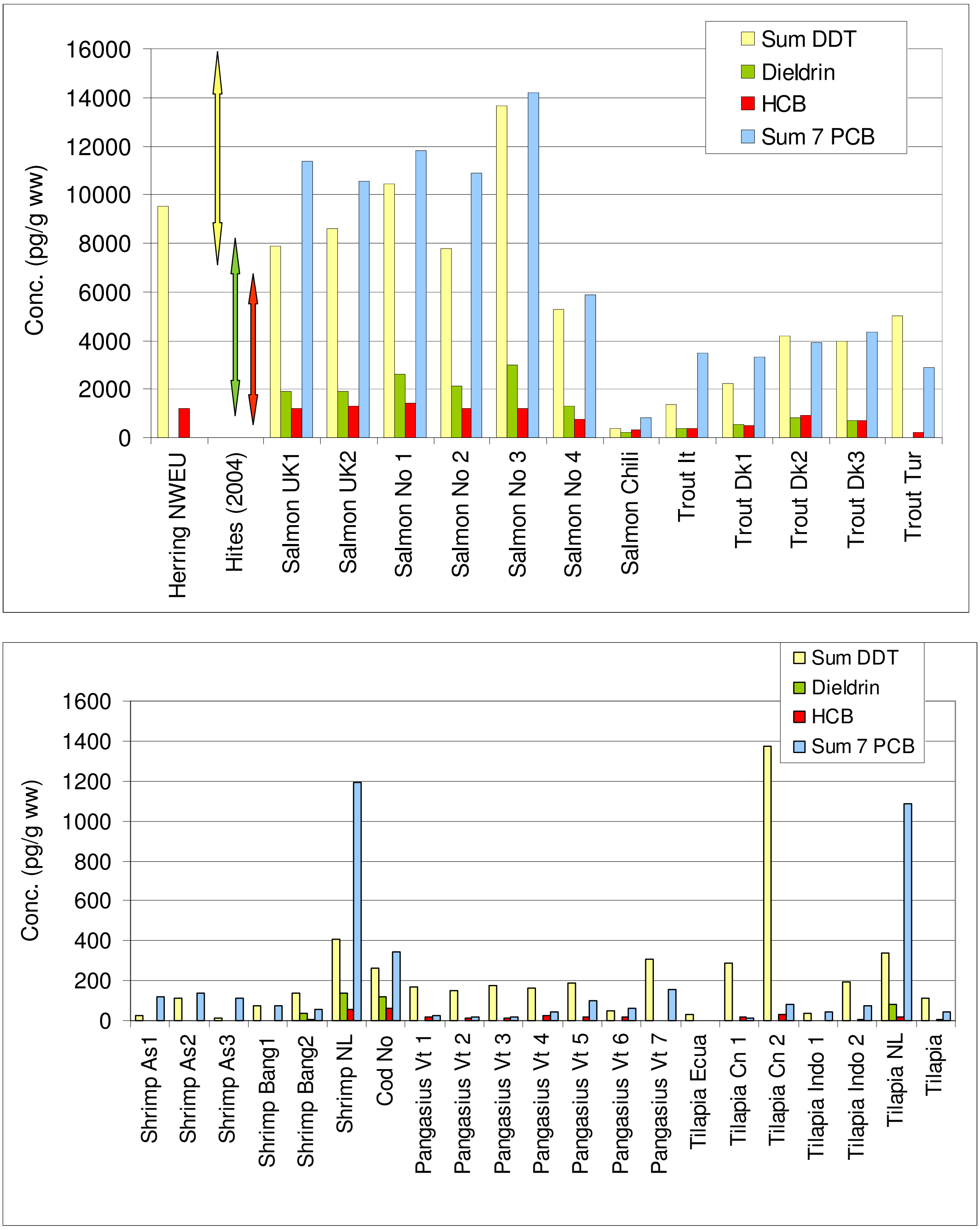


Figure 3.2. *PCB and OCP concentrations in farmed fish and shrimps. In the top graph, data from two other studies are shown for comparison reasons (wild caught herring from North-West Europe (NWEU) (van Leeuwen et al., 2007) and the ranges of salmon samples from the Hites et al. study (Hites et al., 2004a), Country codes: UK = Scotland, No = Norway, It = Italy, Dk = Denmark, Tur = Turkey, As = Asia mixed origins, Bang = Bangladesh, NL = Netherlands, Vt = Vietnam, Ecua = Ecuador, Cn = China and Indo = Indonesia.*

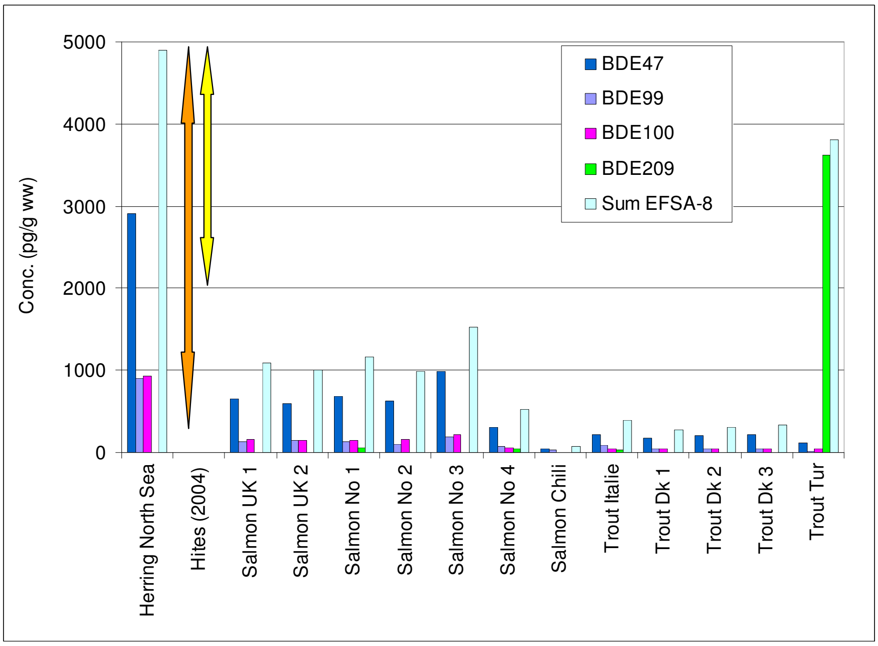
3.1.3 PBDEs

Figure 3.3 shows the BDE 47, 99, 100 and 209 concentrations in salmon and trout (top) and shrimp, cod, pangasius and tilapia (bottom). The EFSA recently recommended monitoring of eight BDEs (28, 47, 99, 100, 153, 154, 183 and 209) (EFSA, 2006), and therefore, the sum of these congeners is also shown ("EFSA-8"). The individual concentrations of all analysed BDEs, as well as the total of all BDEs are given in Appendix 2.

The sum EFSA-8 levels range from 100 to approx 1500 pg/g ww for salmon and trout and <10 to 160 pg/g ww for shrimp, cod, tilapia and pangasius. BDE 47, 99 and 100 are the predominant isomers in salmon, trout (except the Turkish sample) and most tilapia samples. The predominance of these three congeners is commonly observed in wild and farmed fish samples (van Leeuwen and de Boer, 2008; Ashizuka et al., 2008). In smoked trout, a high BDE 209 level was observed. A reason for this is not known. Re-analysis of the sample confirmed the elevated BDE 209 level. Possibly the smoking has played a role, although the smoked salmon (07/870) sample did not show such a profile.

In shrimp and most pangasius, BDE 209 was the predominant congener. The presence of BDE 209 in fish samples was only recently reported, as before it was believed that BDE 209 was not bioavailable (due to its large molecular size and extreme hydrophobicity). The limited quality assurance in some/most laboratories hampered a detection of BDE 209 at very low levels, BDE 209 concentrations reported until now were doubtful. However, Ashizuka et al. (Ashizuka et al., 2008) recently also reported BDE 209 as the predominant congener in two out of three shrimp samples. They speculated that BDE 209 in particulate matter present in the digestive tract caused these elevated BDE 209 levels. Possibly, this plays a role in this study as well. The presence (and predominance) of BDE 209 in pangasius has not been shown before. The reason for this is presently unknown as the feed and local farming conditions were not investigated. BDE 209 was also observed in several fish from Japan, including farmed seabream (Ashizuka et al., 2008). Further research is needed to elucidate the exact causes.

There are no EU MRLs for BDEs in fish and shrimps. In an earlier Dutch study, 2 salmon samples were analysed, of which the Norwegian showed lower levels as observed here, whereas the Scottish sample was approximately 3 times higher (van Leeuwen and de Boer, 2008). The levels in salmon in this study are lower than the range for European salmon reported by Hites et al. in 2004 (Hites et al., 2004b) (Figure 3.3 top, yellow arrow). This suggests that aquaculture industries have put efforts in reducing the BDE levels in feed and, ultimately, in salmon. Compared to wild fish, levels for e.g. BDE 47 in salmon are lower than those observed in herring, but slightly higher than in lean marine fish. The levels in the other farmed fish in this study are (much) lower than those observed in herring and also lean marine fish (and very far below the levels in freshwater fish) (van Leeuwen and de Boer, 2008). Finally, it should be noted that the sum BDE levels are approximately 10 times lower than the sum 7 PCB and the sum DDT levels in the same samples.



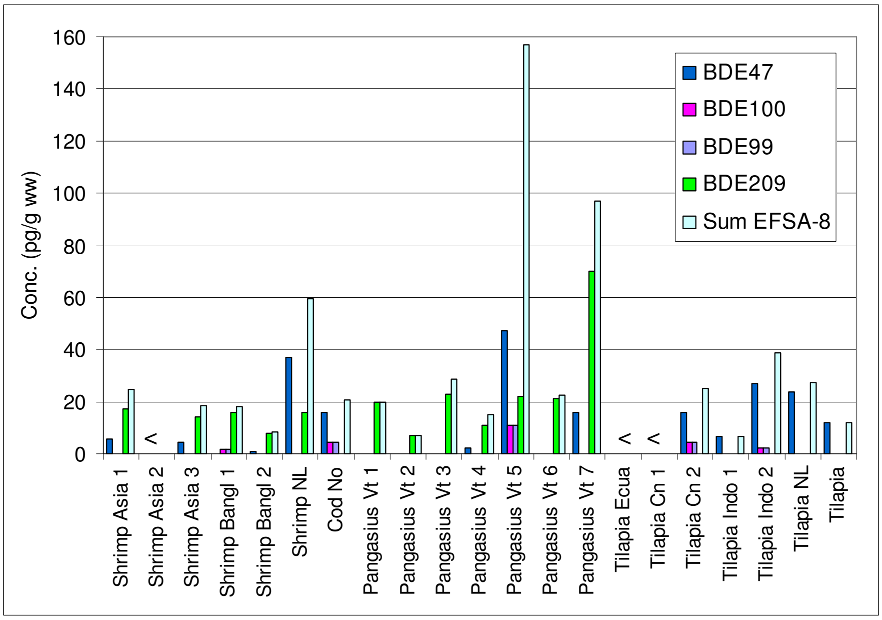


Figure 3.3. PBDEs in farmed fish sampled. In the top graph, salmon and trout are presented, whereas in the lower graph, data on shrimps, cod, pangasius and tilapia are presented. Country codes: UK = Scotland, No = Norway, Dk = Denmark, Tur = Turkey, Asia = Asia mixed origins, Bangl = Bangladesh, NL = Netherlands, Vt = Vietnam, Ecua = Ecuador, Cn = China and Indo = Indonesia. In the sample indicated with < all BDEs were below LOD. For comparison reasons, wild herring from North West Europe (NWEU) (van Leeuwen and de Boer, 2008) are shown. The farmed salmon results from the Hites et al study (Hites et al., 2004b) are shown in orange (Chili) and yellow (Europe) (range of reported results for the sum of 43 congeners). Individual sample results are shown in Appendix 2.

3.1.4 HBCDs

HBCD was detected in 16 samples, ranging from 6 pg/g to 1200 pg/g ww (Dutch shrimps). In all cases, α-HBCD was the predominant diastereomer. β-HBCD was only found in a few samples. The concentrations in tilapia, most pangasius, cod, some shrimp and salmon from Chile were <LOQ (0.01-0.1 ng/g ww for α-HBCD). In an earlier study, HBCD levels in salmon were <0.1 ng/g (Norway) and 1.3 (Scotland) (van Leeuwen and de Boer, 2008). When compared to wild freshwater fish, the levels in the present study are much lower than those in Dutch eel (van Leeuwen and de Boer, 2008). The HBCD levels are also lower than those in wild herring (see Figure 3.4) and mackerel. HBCD levels in lean marine fish (wild) were <0.1 ng/g ww (van Leeuwen and de Boer, 2008).

One farmed shrimp sample (NL) stands out for the high HBCD concentration and the relatively high amounts of β-HBCD (see Figure 3.4). This sample was sent by courier to our laboratory in a polystyrene box, with the shrimps in direct contact with the box. HBCD is commonly applied in polystyrene (although not in polystyrene for food purposes) and HBCD might have been applied as a flame retardant in this box as well. However, it should be noted that in HBCD technical mixtures the γ-HBCD predominates (70-90%), but this diastereomer was not detected in the sample at all. No other data is available on HBCD diastereomers in farmed shrimps and it therefore remains unclear what caused the elevated levels and the specific profile in this sample.

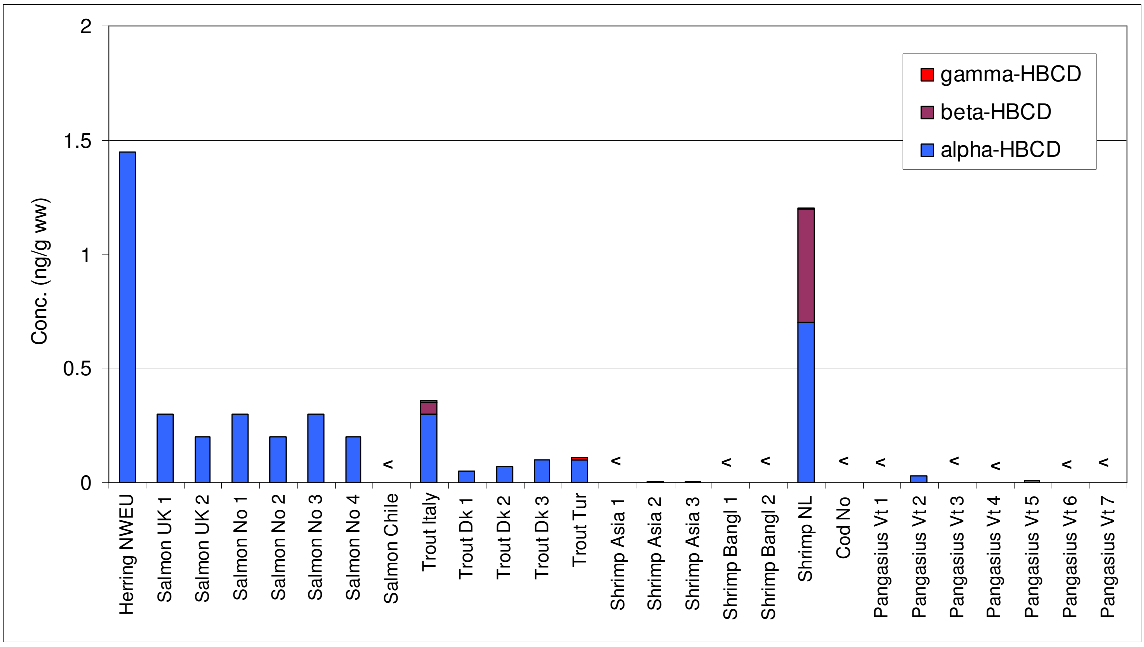


Figure 3.4. *HBCDs in farmed fish samples. In the sample indicated with < all HBCD diastereomers were below LOD and because HBCDs in all tilapia samples were < LOQ, they are not shown in the graph. Wild herring from North West Europe (NWEU) is shown for comparison reasons (van Leeuwen and de Boer, 2008). Country codes: UK: United Kingdom (Scotland), No = Norway, Dk = Denmark, Tur = Turkey, Asia = Asia mixed origins, Bangl = Bangladesh, NL = Netherlands, Vt = Vietnam, Ecua = Ecuador, Cn = China and Indo = Indonesia. Individual sample results are shown in Appendix 2.*

3.1.5 PFCs

In most of the samples, PFCs were not detected at all. Out of all PFC observations (33 samples x 13 PFCs analysed), 41 values were above the LOD (approx. 10%). In these samples PFC values ranged from 0.01 to 0.6 ng/g ww (see Figure 3.5), with PFOS being the isomer detected at highest concentrations. The levels in all other PFCs were (much) lower. It is not commonly observed that PFUnA and PFTrA are detected at higher frequency (in up to 17 samples) than PFOS, and the reason for this is not known (it is randomly observed and not associated with a specific species). It should be noted that the low PFUnA and PFTrA levels challenge the analytical chemists (numbers reported in literature in wild fish are often 1-2 orders of magnitude higher). Uncertainties in these concentrations may therefore be higher (ca. 50% or more). Nevertheless, the QA results (blanks, recoveries, results of the laboratory reference material) give no reason for doubting the results. PFCs accumulate through different mechanisms as lipophilic contaminants. Therefore, salmon and trout do not show the highest PFC contamination levels compared to other species, as was found for compounds like PCBs, OCPs, PBDEs and HBCD.

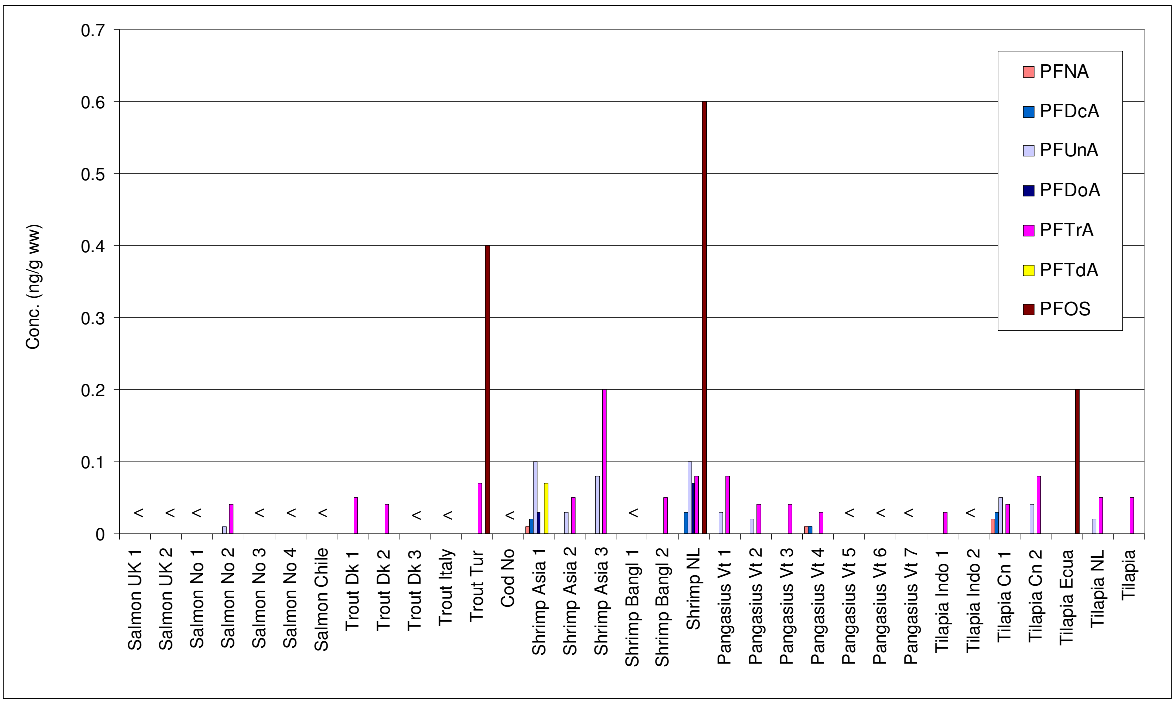


Figure 3.5. PFCs in farmed fish and shrimp samples. Country codes: UK: United Kingdom (Scotland), No = Norway, Dk = Denmark, Tur = Turkey, Asia = Asia mixed origins, Bangl = Bangladesh, NL = Netherlands, Vt = Vietnam, Ecua = Ecuador, Cn = China and Indo = Indonesia. In the sample indicated with < the PFC were below LOD. Individual sample results are shown in Appendix 2.

In an earlier survey on PFCs in wild Dutch freshwater fish, PFOS levels ranged from 5.9 – 150 ng/g ww (eel and pike-perch), whereas levels in wild marine fish levels were <1 to 51 ng/g ww (van Leeuwen and de Boer, 2006). PFOS accumulates in liver tissue, but this study was limited to the fillets of farmed fish. Other PFCs detected in Dutch wild fish (livers) are PFOA, PFNA, PFUnA, PFDoA and PFHxS (van Leeuwen and de Boer, 2006). Levels in Chinese seafood (wild fish) were higher than those observed in this study (for PFOS 0.4-2.9 ng/g ww (fish) and 1.8-14 ng/g ww (shrimp), for PFOA 0.42-0.45 ng/g ww (shrimp) and <LOD (fish), for PFNA all <LOD, for PFDcA 0.3 ng/g ww (shrimp) and <LOD for fish, for PFUnA 0.35-0.65 ng/g ww (fish) and 0.42-0.93 ng/g ww (shrimps)) (Gulkowska et al., 2006). PFOS levels in seafood (wild fish) from Cataluna, Spain were also higher (0.65 ng/g ww), whereas PFHpA and PFOA were also <LOQ (Ericson et al., 2008).

3.1.6 Heavy metals

The heavy metals results are shown in Figure 3.6. Only trout, cod, pangasius and tilapia were analysed for metals, whereas considerable literature data showed that heavy metals in salmon and shrimps were below the EU MRLs. The samples analysed in this study also all meet the EU MRLs for mercury, lead and cadmium in fish (EC, 2002). Lead was below the method LOQ in all samples (<14 to <29 ng/g ww). The mercury levels in trout samples are in the same order as those observed in farmed salmon and eel samples an earlier study (van Leeuwen et al., 2006) and similar to the levels found in the literature survey (van Leeuwen et al., 2008a). The mercury levels in tilapia and pangasius are at the lower end of the levels for salmon, trout and eel in the literature survey. Lead levels in Belgian farmed fish (mostly trout, sampled 2005 and 2006) were approx. 0.02 µg/g ww (Wetenschappelijk Comite van het Federaal Agentschap voor de Veiligheid van de Voedselketen, 2008). The cadmium levels in trout in the current study were slightly lower (only one trout sample contained a detectable cadmium concentration (5 ng/g ww) than those in Belgian farmed fish (mostly trout, <0.01-0.01 µg/g).

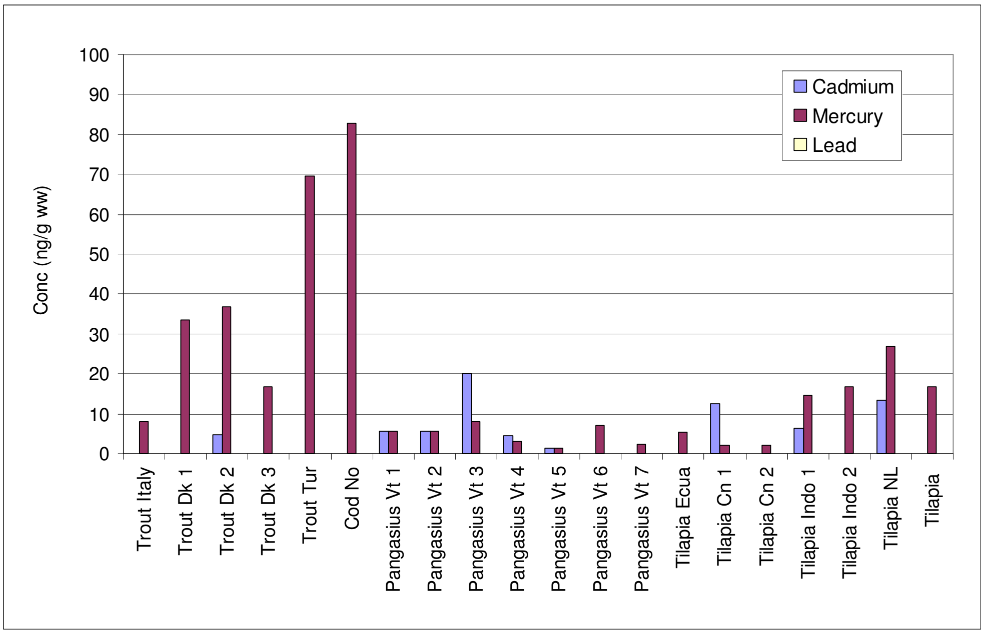


Figure 3.6. Heavy metals in a selection of farmed fish samples (trout, cod, pangasius and tilapia). Country codes: Dk = Denmark, No = Norway, Tur = Turkey, NL = Netherlands, Vt = Vietnam, Ecua = Ecuador, Cn = China and Indo = Indonesia. Lead was in all samples below LOQ (LOQ ranged from 14-29 ng/g ww). Individual sample results are shown in Appendix 2.

3.1.7 Antibiotics

Residues of antibiotics are given in Table 3.1 Screening showed that most samples were negative (below the relevant MRLs), apart from some pangasius and shrimp samples that were positive for macrolides and sulfonamides (see Appendix 2 for detailed sample information). Therefore, confirmation analyses were performed on these samples, which, however, showed that these samples were negative (the initial positive result turned out to be false positive).

In a Belgian study on trout farmed in Belgium in 2004-2006, farmed trout samples were investigated. In a limited number of samples, the nitrofurane levels (incl. metabolites) were above the EU MRL, but all samples were negative for chloramphenicol (Wetenschappelijk Comite van het Federaal Agentschap voor de Veiligheid van de Voedselketen, 2008).

Table 3.1. Aggregated results for the microbiological screening and instrumental analysis of antibiotics. Individual sample results are shown in Appendix 2.

	Sulfonamides	Tetracyclines	Quinolones	Macrolides	Beta-lactams	Nitrofuranes	Chloro-amphenicol
Method	Microbiological screening					LC-MS/MS	LC-MS
Confirmation	LC-UV	N.a.	N.a.	LC-MS	Penase-microb.	N.a.	N.a.
Salmon (n=6)	-	-	-	-	-	-	-
Trout (n=5)	-	-	-	-	-	-	-
Cod (n=1)	-	-	-	-	-	-	-
Pangasius (n=4)	-	-	-	-	-	-	-
Tilapia (n=4)	-	-	-	-	-	-	-
Shrimps (n=5)	-	-	-	-	-	-	-

N.a.: Not applicable

3.2 Contaminant concentrations in fish feed and feed ingredients

3.2.1 Toxaphene

Toxaphene congeners CHB 26, 50 and 62 were analysed in fish feeds and feed ingredients. Toxaphene was found in all samples from animal origin (fish oils, fish meals) and feed containing ingredients from animal origins. Levels in vegetable oils were, as expected, below the LOQ in all samples. Toxaphene is typically found in samples from animal origin. Therefore, vegetable protein and fiber sources were not investigated. CHB 50 was predominant, followed by CHB 26 and CHB 62. The toxaphene levels in all samples meet the current EU MRL for feed (EC, 2005). These MRLs are (for the sum of the three congeners): 0.05 mg/kg (=50 ng/g) for fish feed, 0.2 mg/kg (=200 ng/g) for fish oil and 0.02 mg/kg (=20 ng/g) for fish meal (EC, 2005). Frequent monitoring of feeds may therefore not be of high priority. The directive mentions that the MRLs would be revised by early 2008, with the intention to reduce the MRLs (EC, 2005). However, this revision has not taken place yet.

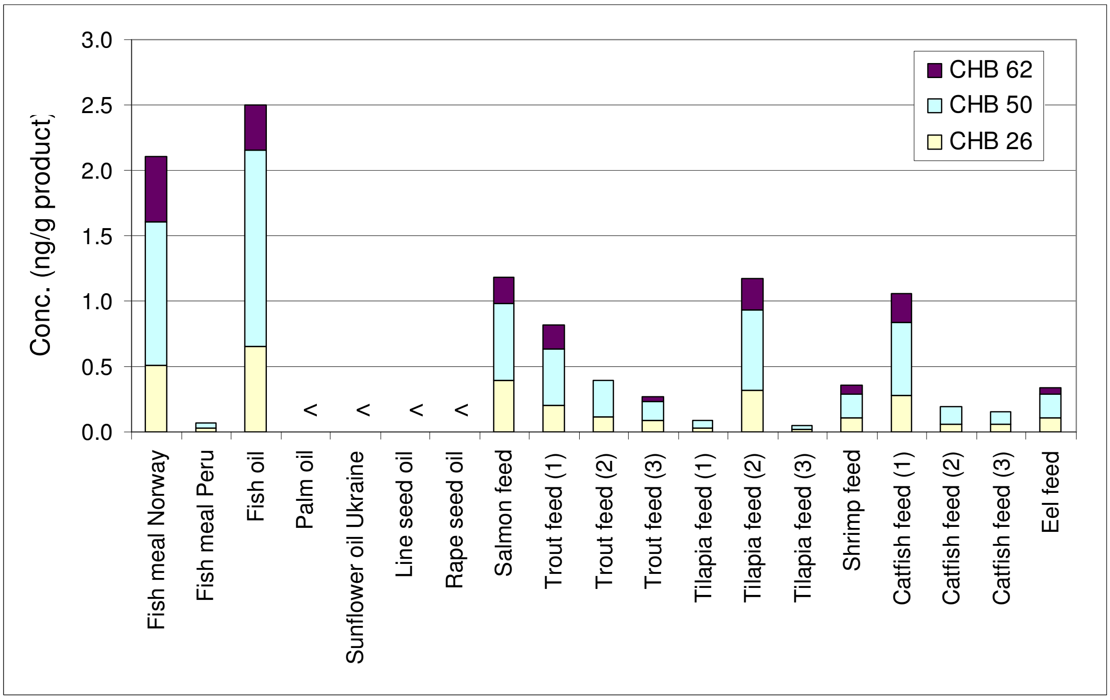


Figure 3.7. Toxaphene concentrations (CHB 26, 50 and 62) in fish feed and feed ingredients. Samples marked by < were below LOD for all three congeners. Individual sample results are shown in Appendix 3.

3.2.2 Mycotoxins

A broad selection of mycotoxins (see Table 2.5) was analysed in fish feeds and feed ingredients. In the ingredients (Figure 3.8, top), the highest levels were observed for fumonisins B1, B2 and B3 in corn, whereas these were not observed in wheat and soy products, which is a common phenomenon for these matrices. DON was observed both in corn and wheat. Aflatoxin B1, ergotamin, T2- and HT2-toxin, alternariol and ochratoxin A were only found at few occasions, of which the high level of ergotamin in one sample is most remarkable. No mycotoxins were found in Dutch rapeseed, pea meal, palm oil and soy products (except soybean meal). Four fish meal and fish oils were also investigated. Only in one fish meal sample, 90 ng/g DON was detected, being much lower than the levels observed in vegetable protein sources. A legislative limit only exists for Aflatoxine B1 in feed (0.01 mg/kg) (EC, 2003), and all feed samples were well below this limit. In 2006, guidance values were published by the European Commission on DON, ZEN ochratoxin A and fumonisin B1+B2 in feed materials and feeds (EC, 2006b). All ingredient and feed samples (to which these limits apply) were well below the guidance values.

Concerning feeds, highest levels were observed in feeds for herbivorous and omnivorous fish and no mycotoxins were found in feed for salmon. DON and ZEN were observed in nearly all samples, and the fumonisins were observed in 3 samples only, suggesting that corn or corn gluten were used as ingredients in these feeds. Mycotoxin levels as measured in this study are comparable to reported ones in literature.

Bintvihok et al. (Bintvihok et al., 2003) reported levels below 1 ng/g in shrimp feed. They also studied the effect of fed diets containing up to 20 ng/g aflatoxin B1, to find out that there was a slight mortality effect at the highest contamination level. An older study by Bautista et al. (Bautista et al., 1994) concluded that shrimp growth decreased at aflatoxin B1 levels above 50 ng/g. Tuan et al. (Tuan et al., 2002) carried out a comparable study in tilapia. The level at which growth was influenced was found to be 250 ng/g, which is more then 10 times higher. None of these studies reported aflatoxin levels in the shrimp or fish tissue. Carlson et al. (Carlson et al., 2001) investigated the influence of fumonisin on trout, which was aflatoxin B1 initiated at a level of 100 ng/g. They measured an increase of liver cancers above 23 µg/g of fumonisin at this background aflatoxin B1 level. Burgos-Hernandez et al. (Burgos-Hernandez et al., 2005) reported similar conclusions in shrimp. Trigo-Stockli et al. (Trigo-Stockli et al., 2000) reported that DON concentrations above 1 µg/g led to significant reductions in growth and body weight. But even at the 1 µg/g level in feed, they did not detect DON in shrimp.

The sensitivity for mycotoxins differs for any marine species. Manning et al. reported catfish to tolerate more dietary DON intake, whereas it seemed more susceptible to T2-toxin (Manning et al., 2003a). Levels as low as 625 ng/g reduced catfish weight gain and higher concentrations (2500 ng/g) significantly reduced feed conversion and survival rate. The latter level was comparable for a study on ochratoxin A, where 2000 ng/g was the level above which these effects were observed (Manning et al., 2003b). For the same catfish species Yildirim et al. (Yildirim et al., 2000) investigated the effect of fumonisin and moniliformin, separately and in combination. For these two mycotoxins the level at which loss of weight was observed, was above 20,000 ng/g. Combination of fumonisin and moniliformin in one diet, did only slightly affect the livers of the catfish. The same mycotoxins and its combination were investigated by Tuan et al. (Tuan et al., 2003) in Nile tilapia, where the level of these 2 mycotoxins could be 2 times higher to decrease growth. For zearalenone Arukwe et al. (Arukwe et al., 1999) reported that at a level of 1 mg/kg could affect reproducibility and the development of fish eggs.

All mycotoxin levels in these studies are far above the ones as reported in this study. The levels in these studies were expressed as µg/g in feed, whereas ng/g levels are measured in this study. Levels in the fish or shrimps as such were seldom reported. If so, they were below the limit of determination, which is comparable with the few data as given here for shrimp (see Appendix 3 for the levels in selected shrimp samples). It is currently not known to what extent this holds for fish samples. Regarding the literature data it is clear that effects depend on the type of mycotoxin and the fish species. Differences in species susceptibility have not yet been investigated so extensively. The data in this study suggest that when the level in feed is low enough, no contamination could be expected in the edible product. For a more accurate risk analysis it would be helpful to do more measurements on this topic.

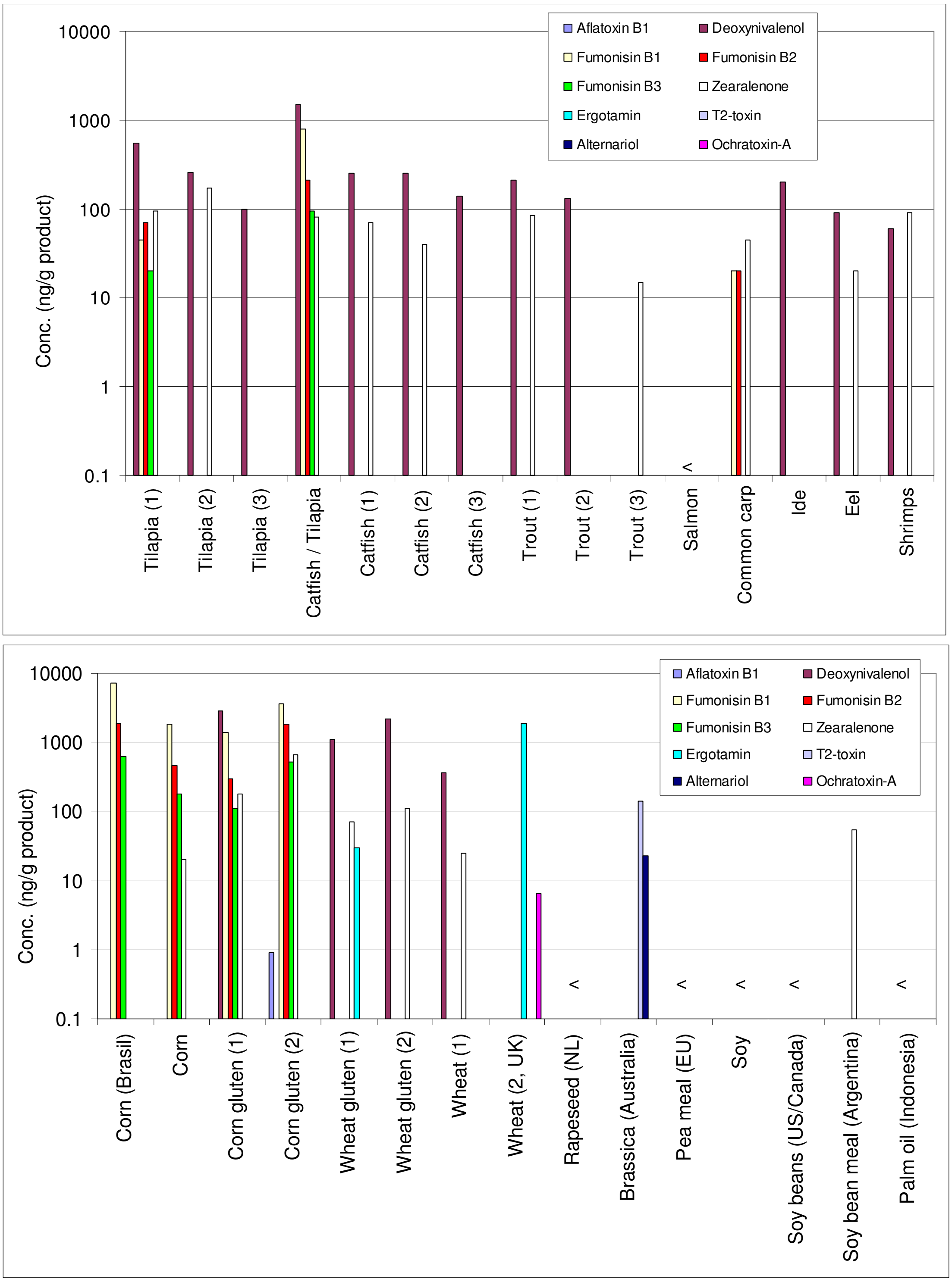


Figure 3.8. Levels of mycotoxins in feed ingredients (top) and fish feeds (bottom). The mycotoxins with detectable levels are shown, whereas the remaining mycotoxins (see Table 2.4) were all below LOQ. Further details can be found in Appendix 3

4. Conclusions

This extensive study shows that contaminant levels in the most popular Dutch farmed fish (salmon, trout, tilapia, pangasius) and farmed shrimps are very low (mostly < 1 ng/g ww), and far below the applicable legislative limits. The contaminant levels decrease in the following order PCBs ≈ OCPs > HBCD ≈ PBDEs ≈ PFCs > PCDD/Fs and dl-PCBs. Generally, the order of concentration decreases in the following order: salmon > trout > tilapia ≈ pangasius ≈ shrimp. Levels in farmed fish are generally lower than levels observed in wild fish. The levels of heavy metals were also well below the applicable legislative limits.

Toxaphene levels in fish feed and feed ingredients (sum of CHB 26, 50 and 62) are well below the applicable legislative limits. Fumonisins, ZEN and DON were regularly detected in feed ingredients and fish feeds although the levels stayed well below legislative and guidance limits. More research is needed to investigate to what extent mycotoxins accumulate into fish.

The Dutch consumption of farmed fish is increasing, and within this group, the share of pangasius and tilapia is growing rapidly. Considering the low contaminant levels observed in pangasius and tilapia, it is believed that the human exposure to PCBs, OCPs, PBDEs, HBCD, PFCs, PCDD/Fs and dl-PCBs through fish consumption will further decrease

5. Recommendations

In general the levels of all the investigated contaminants in popular farmed fish consumed in The Netherlands are well below the level of contamination in various wild fish. Therefore, there is little need to intensify the regular monitoring of these contaminants in farmed fish.

Mycotoxins were detected in aquaculture feeds and ingredients. It is therefore recommended to determine if and to what extent mycotoxins are transferred to the fillets of fish species, and if this is a relevant exposure pathway for humans. Preliminary analysis of six shrimp samples revealed no detectable levels of mycotoxins, but it should be noted that no details of mycotoxins levels in their diets are available.

6. Acknowledgements

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Appendix I. Sample details

Table I.1 Sample details.

IVM LIMS code	Species	Latin name	Country of origin	No indi-viduals in pooled sam-ple	Weigt of pooled sam-ple (gram)	Whole fish , whole fillet or piece of a fillet*	Physical state at pur-chase
07/860	Salmon	Salmo salar	Norway	2	1107	Fillet (piece)	4°C, raw
07/790	Salmon	Salmo salar	Norway	10	941	Fillet (piece), skin removed	4°C, raw
07/804	Salmon	Salmo salar	Norway	5	491	Fillet (piece)	4°C, raw
07/870	Salmon	Salmo salar	Norway	10	939	Fillet (piece)	4°C, smoked
07/805	Salmon	Salmo salar	UK (Scotland)	5	480	Fillet (piece)	4°C, raw
07/785	Salmon	Salmo salar	UK (Scotland)	10	1441	Fillet (piece)	4°C, raw
07/810	Salmon	Salmo salar	Chile	10	1213	Fillet (piece)	-20°C, raw
07/862	Trout	N.a.	Denmark	10	1359	Whole fish, filleted	4°C, raw
07/861	Trout	N.a.	Denmark	10	1208	Fillet	4°C, raw
07/807	Trout	Onchorhynchus mykiss	Denmark	5	296	Fillet	4°C, raw
07/793	Trout	Salmo trutta	Italy	10	1081	Whole fish, filleted	4°C, raw
07/869	Trout	Onchorhynchus mykiss	Turkey	10	949	Fillet (whole)	4°C, smoked
07/787	Cod	Gadus Morhua	Norway	10	1189	Fillet (piece)	4°C, raw
07/864	Pangasius	Pangasius hypophthalmus	Vietnam	10	1383	Fillet (whole)	4°C, raw

IVM LIMS code	Species	Latin name	Country of origin	No individuals in pooled sample	Weigt of pooled sample (gram)	Whole fish , whole fillet or piece of a fillet*	Physical state at purchase
08/002	Pangasius	Pangasius hypophthalmus	Vietnam	6	Nr.	Fillet (whole)	4°C, raw
07/868	Pangasius	Pangasius hypophthalmus	Vietnam	8	1013	Fillet (whole)	-20°C, raw
07/791	Pangasius	Pangasius hypophthalmus	Vietnam	10	1187	Fillet (whole)	4°C, raw
07/796	Pangasius	Pangasius hypophthalmus	Vietnam	Approx. 12	1594	Fillet (whole)	-20°C, raw
07/806	Pangasius	Pangasius hypophthalmus	Vietnam	5	870	Fillet (whole)	-20°C, raw
07/786	Pangasius	Pangasius hypophthalmus	Vietnam	9	372	Fillet (whole)	4°C, raw
07/809	Shrimps	Penaeus monoden	Bangladesh	41/50**	827	N.a.	-20°C, raw
07/797	Shrimps	Penaeus monoden	Bangladesh	21-30/kg**	867	Whole, <u>heads removed</u>	-20°C, raw, unpeeled
07/811	Shrimps	Litopenaeus vannamei	Indonesia/China	45/50**	1275	Whole	-20°C, cooked
07/794	Shrimps	Penaeus monoden	Bangladesh / India	N.r.	1444	N.a.	4°C, blanched
07/789	Shrimps	Litopenaeus vannamei	Thailand, Malaysia & Indonesia	N.r.	787	N.a.	4°C, cooked
07/871	Shrimps	N.a.	Netherlands	30/kg**	871	Whole, <u>heads removed</u>	4C, raw, unpeeled
07/863	Tilapia	Oreochromis spp	N.r.	10	1654	Fillet (whole)	4°C, raw
07/792	Tilapia	Oreochromis mossambica	China	10	1149	Fillet (whole)	4°C, raw
07/808	Tilapia	Oreochromis nilloticus	China	6	956	Fillet (whole)	-20°C, raw
07/788	Tilapia	Oreochromis nilloticus	Ecuador	10	985	Fillet (whole)	4°C, raw
07/795	Tilapia	Oreochromis spp	Indonesia	Approx. 18	1649	Fillet (whole)	-20°C, raw

IVM LIMS code	Species	Latin name	Country of origin	No individuals in pooled sample	Weigt of pooled sample (gram)	Whole fish , whole fillet or piece of a fillet*	Physical state at purchase
08/001	Tilapia	Oreochromis niloticus	Indonesia	6	N.r.	Fillet (whole)	4°C, raw
07/867	Tilapia	Oreochromis spp	Indonesia	Approx. 18	1578	Fillet (whole)	-20°C, raw

N.a.: not applicable; N.r.: not recorded.

* Underlined: additional pre-treatment prior to grinding and homogenisation of pooled sample

** Declared sizes (e.g. in no of individuals/kg)

Appendix II. Detailed information on contaminants levels in farmed fish samples

Table II.1. Levels of PCDD/Fs and dl-PCBs in selected farmed fish samples (pg TEQ/g ww).

			Lowerbound			Upperbound		
RIKILT LIMS	IVM LIMS	Sample type and origin	dl-PCBs	PCDD/Fs	Total-TEQ	dl-PCBs	PCDD/Fs	Total-TEQ
208198	786	Pangasius Vietnam 1	0.00	0.00	0.00	0.02	0.17	0.19
208201	791	Pangasius Vietnam 2	0.00	0.01	0.01	0.02	0.18	0.20
208204	796	Pangasius Vietnam 3	0.00	0.00	0.00	0.02	0.17	0.19
208206	806	Pangasius Vietnam 4	0.00	0.00	0.00	0.02	0.17	0.19
208210	864	Pangasius Vietnam 5	0.00	0.00	0.00	0.02	0.17	0.19
208212	868	Pangasius Vietnam 6	0.00	0.00	0.00	0.02	0.17	0.19
208203	795	Tilapia Indonesia 1	0.01	0.00	0.01	0.02	0.17	0.19
208211	867	Tilapia Indonesia 2	0.00	0.00	0.00	0.02	0.17	0.19
208207	808	Tilapia China 2	0.01	0.00	0.01	0.03	0.17	0.20
208200	788	Tilapia Ecuador	0.00	0.00	0.00	0.02	0.17	0.19
208209	863	Tilapia	0.01	0.00	0.01	0.03	0.17	0.20
208199	787	Cod Norway	0.02	0.00	0.02	0.04	0.17	0.21
208205	797	Shrimp Bangladesh 1	0.01	0.01	0.01	0.03	0.17	0.20
208202	794	Shrimp Asia 2	0.01	0.00	0.01	0.03	0.17	0.20
208208	811	Shrimp Asia 3	0.01	0.00	0.01	0.03	0.17	0.20
208213	871	Shrimp Netherlands	0.08	0.00	0.08	0.09	0.17	0.26

Table II.2. Levels of PCBs in farmed fish samples (pg/g ww).

IVM LIMS	Sample type and origin	PCB 28	PCB 52	PCB 101	PCB 118	PCB 138	PCB 153	PCB 180	Som PCBs
07/0785	Salmon Scotland (UK) 1	<100	970	1100	1700	3000	3500	1100	11370
07/0805	Salmon Scotland (UK) 2	<150	700	740	1500	2900	3500	1200	10540
07/0790	Salmon Norway 1	<130	1000	1100	1800	3100	3700	1100	11800
07/0804	Salmon Norway 2	<150	1000	760	1500	3000	3500	1100	10860
07/860	Salmon Norway 3	450	960	1900	1700	3400	4500	1300	14210
07/870	Salmon Norway 4	230	480	770	770	1500	1800	570	6120
07/0810	Salmon Chile	<86	<86	<86	<86	350	320	170	840
07/0793	Trout Italy	190	590	310	610	790	750	240	3480
07/807	Trout Denmark 1	120	250	510	420	880	900	260	3340
07/861	Trout Denmark 2	180	340	600	470	890	1100	320	3900
07/862	Trout Denmark 3	170	310	590	530	1100	1300	340	4340
07/869	Trout Turkey	110	180	350	360	700	860	300	2860
07/0789	Shrimp Asia 1	<13	<13	<13	32	41	44	<13	117
07/0794	Shrimp Asia 2	<6.7	<6.7	78	12	19	18	10	137
07/0811	Shrimp Asia 3	<11	<11	<11	28	36	37	15	116
07/0797	Shrimp Bangladesh 1	<11	<11	65	<11	<11	11	<11	76
07/809	Shrimp Bangladesh 2	9.2	6.3	7.6	8	10	11	5.1	57
07/871	Shrimp Netherlands	35	18	150	190	370	350	78	1191
07/787	Cod Norway	<8.4	32	22	53	99	110	28	344

IVM LIMS	Sample type and origin	PCB 28	PCB 52	PCB 101	PCB 118	PCB 138	PCB 153	PCB 180	Som PCBs
07/786	Pangasius Vietnam 1	<42	13	<6.9	<6.9	10	<6.9	<6.9	23
07/791	Pangasius Vietnam 2	<30	6.9	<5	<5	8.8	5.6	<5	21
07/796	Pangasius Vietnam 3	<14	5	<2.4	2.4	5.8	4.4	<2.4	18
07/806	Pangasius Vietnam 4	<17	10	6.3	<5.4	11	12	7.6	47
07/864	Pangasius Vietnam 5	31	20	9.9	<6.7	15	19	8	103
07/868	Pangasius Vietnam 6	21	15	9.2	<5.1	9.4	11	<5.1	66
08/002	Pangasius Vietnam 7	<140	59	<23	<23	41	60	<23	160
07/788	Tilapia Ecuador	<42	<7	<7	<7	<7	<7	<7	N.a.
07/792	Tilapia China 1	<30	6.5	7.1	<5	<5	<5	<5	14
07/808	Tilapia China 2	<39	14	<6.6	12	22	20	16	84
07/795	Tilapia Indonesia 1	<21	15	<6.8	<6.8	14	18	<6.8	47
07/867	Tilapia Indonesia 2	<48	13	<7.6	<7.6	22	29	11	75
08/001	Tilapia Netherlands	<21	57	110	130	280	370	140	1087
07/863	Tilapia	<40	8.9	<6.6	<6.6	21	15	<6.6	45

N.a.: Not applicable

Table II.3. Levels of OCPs in farmed fish samples (pg/g ww).

IVM LIMS	Sample type and origin	OCPs*											
		HCBD	QCB	HCB	α-HCH	β-HCH	γ-HCH	Heptachlor	Aldrin	Telodrin	Isodrin	Dieldrin	Endrin
07/0785	Salmon Scotland (UK) 1	<34	<34	1200	<100	<100	<100	<100	<100	<100	<100	1900	<100
07/0805	Salmon Scotland (UK) 2	<50	150	1300	<150	<150	<150	<150	<150	<150	<150	1900	<150
07/0790	Salmon Norway 1	<43	150	1400	<130	<130	<130	<130	<130	<130	<130	2600	<130
07/0804	Salmon Norway 2	<52	<52	1200	<150	<150	<150	<150	<150	<150	<150	2100	<150
07/860	Salmon Norway 3	<11	150	1200	<38	<38	<38	<22	<22	<38	<22	3000	<38
07/870	Salmon Norway 4	<12	100	750	<43	<43	<43	<24	<24	<43	<24	1300	<43
07/0810	Salmon Chile	<29	<29	300	<86	<86	<86	<86	<86	<86	<86	200	<86
07/0793	Trout Italy	<25	<25	390	<76	<76	<76	<76	<76	<76	<76	390	<76
07/807	Trout Denmark 1	<14	98	480	<44	<44	<44	<22	<22	<44	<22	550	<44
07/861	Trout Denmark 2	<6.2	1500	900	<22	<22	<22	<12	<12	<22	<12	820	<22
07/862	Trout Denmark 3	<7.4	6100	710	<26	<26	<26	<15	<15	<26	<15	690	<26
07/869	Trout Turkey	<5.4	32	220	<19	<19	<19	<11	<11	<19	<11	<19	<19
07/0789	Shrimp Asia 1	<4.5	<4.5	<13	<13	<13	<13	<13	<13	<13	<13	<13	<13
07/0794	Shrimp Asia 2	<2.2	<2.2	<6.7	<6.7	<6.7	<6.7	<6.7	<6.7	<6.7	<6.7	<6.7	<6.7
07/0811	Shrimp Asia 3	<3.8	<3.8	<11	<11	<11	<11	<11	<11	<11	<11	<11	<11
07/0797	Shrimp Bangladesh 1	<3.8	<3.8	<11	<11	<11	<11	<11	<11	<11	<11	<11	<11
07/809	Shrimp Bangladesh 2	<1.3	3.8	3.8	<4.3	<4.3	<4.3	<2.1	<2.1	<4.3	<2.1	38	<4.3
07/871	Shrimp Netherlands	<4.5	11	58	<15	<15	<15	<7.3	<7.3	<15	<7.3	140	<15

IVM LIMS	Sample type and origin	OCPs*											
		HCBD	QCB	HCB	α-HCH	β-HCH	γ-HCH	Heptachlor	Aldrin	Telodrin	Isodrin	Dieldrin	Endrin
07/787	Cod Norway	<0.7	5.8	64	<2.4	<2.4	<2.4	<1.4	<1.4	<2.4	<1.4	120	<2.4
07/786	Pangasius Vietnam 1	<3.5	13	17	24	<12	<12	<6.9	<6.9	<12	<6.9	<12	<12
07/791	Pangasius Vietnam 2	<2.5	15	14	<8.7	<8.7	<8.7	<5	<5	<8.7	<5	<8.7	<8.7
07/796	Pangasius Vietnam 3	<1.2	9.7	11	<4.2	<4.2	<4.2	<2.4	<2.4	<4.2	<2.4	<4.2	<4.2
07/806	Pangasius Vietnam 4	<3.3	8.9	28	<11	<11	<11	<5.4	<5.4	<11	<5.4	<11	<11
07/864	Pangasius Vietnam 5	<4.2	<21	18	<13	<13	<13	<6.7	<6.7	<13	<6.7	<13	<13
07/868	Pangasius Vietnam 6	<3.2	17	16	<10	<10	<10	<5.1	<5.1	<10	<5.1	<10	<10
08/002	Pangasius Vietnam 7	<14	<14	<23	<46	<46	<46	<23	<23	<46	<23	<46	<46
07/788	Tilapia Ecuador	<3.5	21	<7	<12	<12	<12	<7	<7	<12	<7	<12	<12
07/792	Tilapia China 1	<2.5	23	20	<8.7	<8.7	<8.7	<5	<5	<8.7	<5	<8.7	<8.7
07/808	Tilapia China 2	<3.3	34	29	50	<12	<12	<6.6	<6.6	<12	<6.6	<12	<12
07/795	Tilapia Indonesia 1	<4.3	14	<6.8	<14	<14	<14	<6.8	<6.8	<14	<6.8	<14	<14
07/867	Tilapia Indonesia 2	<4.8	<24	8.9	<15	<15	<15	<7.6	<7.6	<15	<7.6	<15	<15
08/001	Tilapia Netherlands	<13	<13	21	<42	<42	<42	<21	<21	<42	<21	84	<42
07/863	Tilapia	<3.3	9.6	8	31	<12	<12	<6.6	<6.6	<12	<6.6	<12	<12

*HCBD: hexachlorobutadiene; QCB: pentachlorobenzene; HCB: hexachlorobenzene and HCH: hexachlorocyclohexane

Table II.3 (continued). Levels of OCPs in farmed fish samples (pg/g ww).

IVM LIMS	Sample type and origin	OCPs*									
		α-endosulfan	trans-HEPO	cis-HEPO	o,p'-DDE	p,p'-DDE	o,p'-DDD	p,p'-DDD	o,p'-DDT	p,p'-DDT	Sum DDTs
07/0785	Salmon Scotland (UK) 1	<100	<100	<100	<100	4800	<100	1900	<100	1200	7900
07/0805	Salmon Scotland (UK) 2	<150	<150	<150	<150	3900	<150	2000	720	2000	8620
07/0790	Salmon Norway 1	<130	<130	<130	<130	5300	410	2900	440	1400	10450
07/0804	Salmon Norway 2	<150	<150	<150	<150	4500	<150	2400	<150	890	7790
07/860	Salmon Norway 3	<38	<38	<38	<22	8100	<38	3600	380	1600	13680
07/870	Salmon Norway 4	<43	<43	<43	<24	3300	<43	1400	180	410	5290
07/0810	Salmon Chile	480	<86	<86	<86	110	<86	270	<86	<86	380
07/0793	Trout Italy	<76	<76	<76	<76	540	<76	450	<76	350	1340
07/807	Trout Denmark 1	<44	<44	<44	<22	1400	<44	570	<22	240	2210
07/861	Trout Denmark 2	<22	<22	<22	<12	2200	<22	770	<12	1200	4170
07/862	Trout Denmark 3	<26	<26	<26	<15	2200	<26	1100	<15	680	3980
07/869	Trout Turkey	<19	<19	<19	<11	3500	<19	950	<11	560	5010
07/0789	Shrimp Asia 1	<13	<13	<13	<13	26	<13	<13	<13	<13	26
07/0794	Shrimp Asia 2	<6.7	<6.7	<6.7	<6.7	110	<6.7	<6.7	<6.7	<6.7	110
07/0811	Shrimp Asia 3	<11	<11	<11	<11	11	<11	<11	<11	<11	11
07/0797	Shrimp Bangladesh 1	<11	<11	<11	<11	77	<11	<130	<11	<11	77
07/809	Shrimp Bangladesh 2	<4.3	<4.3	<4.3	<2.1	140	<4.3	<4.3	<2.1	<4.3	140
07/871	Shrimp Netherlands	<15	<15	<15	<7.3	410	<15	<15	<7.3	<15	410

IVM LIMS	Sample type and origin	OCPs*									
		α-endosulfan	trans-HEPO	cis-HEPO	o,p'-DDE	p,p'-DDE	o,p'-DDD	p,p'-DDD	o,p'-DDT	p,p'-DDT	Sum DDTs
07/787	Cod Norway	<2.4	<2.4	<2.4	<1.4	190	<2.4	67	7.9	<2.4	264.9
07/786	Pangasius Vietnam 1	<12	<12	<12	<6.9	59	<12	57	<6.9	51	167
07/791	Pangasius Vietnam 2	<8.7	<8.7	<8.7	<5	53	<8.7	46	12	37	148
07/796	Pangasius Vietnam 3	<4.2	<4.2	<4.2	<2.4	43	<4.2	60	11	61	175
07/806	Pangasius Vietnam 4	<11	<11	<11	<5.4	32	<11	32	57	40	161
07/864	Pangasius Vietnam 5	74	<13	<13	<6.7	60	<13	95	<6.7	34	189
07/868	Pangasius Vietnam 6	<10	<10	<10	<5.1	22	<10	27	<5.1	<10	49
08/002	Pangasius Vietnam 7	<46	<46	<46	<23	130	<46	93	<23	83	306
07/788	Tilapia Ecuador	<12	<12	<12	<7	33	<12	<12	<7	<12	33
07/792	Tilapia China 1	<8.7	<8.7	<8.7	<5	130	<8.7	110	12	35	287
07/808	Tilapia China 2	<12	<12	<12	<6.6	660	<12	660	<6.6	55	1375
07/795	Tilapia Indonesia 1	<14	<14	<14	<6.8	39	<14	<14	<6.8	<14	39
07/867	Tilapia Indonesia 2	<15	<15	<15	<7.6	92	<15	41	61	<15	194
08/001	Tilapia Netherlands	<42	<42	<42	<21	230	<42	110	<21	<42	340
07/863	Tilapia	<12	<12	<12	<6.6	110	<12	<12	<6.6	<12	110

* HEPO: heptachlorepoxide

Table II.4. Levels of PBDEs in farmed fish samples (pg/g ww).

		BDE											
Sample and origin	IVM LIMS	17	28	47	49	66	71	77	85	99	100	119	126
Salmon Scotland (UK) 1	07/785	42	27	650	140	<48	<26	<14	<13	130	160	29	42
Salmon Scotland (UK) 2	07/805	26	28	600	150	<68	<36	<20	<18	140	140	28	30
Salmon Norway 1	07/790	24	26	690	150	<61	<33	<18	<16	130	150	29	37
Salmon Norway 2	07/804	41	34	630	180	<72	<38	<21	<19	100	160	<19	17
Salmon Norway 3	07/860	<27	43	990	260	<150	<38	<25	<23	190	220	43	29
Salmon Norway 4	07/870	<14	<17	310	83	<81	<20	<13	<12	71	65	<18	17
Salmon Chili	07/810	<13	<11	44	<21	<40	<21	<12	<11	31	<11	<11	<6.8
Trout Italie	07/793	<5.2	8.8	220	20	<16	<8.7	<4.9	<4.3	94	37	<4.3	<2.8
Trout Denmark 1	07/807	<5.4	<4.5	170	32	<45	<6	<9	<5.1	38	38	8.7	9.4
Trout Denmark 2	07/861	<7.3	<8.5	200	28	<41	<10	<6.8	<6.1	50	41	<9.3	8.4
Trout Denmark 3	07/862	<8.9	<10	220	51	<50	<12	<8.3	<7.5	49	48	<11	6.5
Trout Tur	07/869	<6.3	<7.4	120	24	<35	<8.8	<5.9	<5.3	7.3	48	<8	<4.1
Shrimp Asia 1	07/789	<1.9	<1.6	5.9	<3.2	<6	<3.2	<1.8	<1.6	<1.6	<1.6	<1.6	<1
Shrimp Asia 2	07/794	<1	<0.83	<1.5	<1.7	<3.1	<1.7	<0.94	<0.83	<0.83	<0.83	<0.83	<0.54
Shrimp Asia 3	07/811	<1.7	<1.4	4.5	<2.8	<5.3	<2.8	<1.6	<1.4	<1.4	<1.4	<1.4	<0.91
Shrimp Bangl 1	07/797	<1.7	<1.4	<2.6	<2.9	<5.4	<2.9	<1.6	<1.4	<1.4	1.9	<1.4	<0.92
Shrimp Bangl 2	07/809	<0.49	<0.4	0.7	<0.97	<4	<0.54	<0.81	<0.46	<0.46	<0.46	<0.4	1.2
Shrimp Netherlands	07/871	<1.8	2	37	<3.6	<15	<2	<3	<1.7	<1.7	<1.7	<1.5	2

		BDE											
Sample and origin	IVM LIMS	17	28	47	49	66	71	77	85	99	100	119	126
Cod Norway	07/787	<1.5	<1.8	16	7.3	<8.5	<2.1	<1.4	N.d.	<1.5	4.5	<1.9	<0.98
Pangasius Vietnam 1	07/786	<4.5	<3.8	<6.8	<7.5	<14	<7.5	<4.2	<3.8	<3.8	<3.8	<3.8	<2.4
Pangasius Vietnam 2	07/791	<3.4	<2.8	<5	<5.6	<10	<5.6	<3.1	<2.8	<2.8	<2.8	<2.8	<1.8
Pangasius Vietnam 3	07/796	<1.4	<1.7	<2.7	<2.3	<8	<2	<1.3	<1.2	5.7	<2	<1.8	<0.92
Pangasius Vietnam 4	07/806	<1.3	<1	2.2	<2.5	<10	<1.4	<2.1	<1.2	2.0	<1.2	1.1	<1.2
Pangasius Vietnam 5	07/864	<1.7	<1.4	47	<3.3	<14	<1.8	<2.8	3.1	60	11	<1.4	<1.5
Pangasius Vietnam 6	07/868	<1.3	<1.1	<1.8	<2.6	<11	<1.5	<2.2	<1.3	1.5	<1.3	<1.1	<1.2
Pangasius Vietnam 7	08/002	<5.7	<4.7	16	<11	<47	<6.3	<9.5	<5.4	11	<5.4	<4.7	<5.3
Tilapia Ecuador	07/788	<4.7	<3.9	<7	<7.8	<15	<7.8	<4.4	<3.9	<3.9	<3.9	<3.9	<2.5
Tilapia China 1	07/792	<3.4	<2.8	<5	<5.6	<11	<5.6	<3.2	<2.8	<2.8	<2.8	<2.8	<1.8
Tilapia China 2	07/808	<4.5	<3.7	16	<7.5	<14	<7.5	<4.2	<3.7	<3.7	4.2	<3.7	3.8
Tilapia Indonesia 1	07/795	<1.7	<1.4	6.5	<3.5	<14	<1.9	<2.9	<1.7	<1.7	<1.7	<1.4	<1.6
Tilapia Indonesia 2	07/867	<1.9	3.0	27	<3.7	<16	<2.1	<3.1	<1.8	2.1	2.3	<1.6	<1.7
Tilapia Netherlands	08/001	<5.1	<4.2	24	<10	<42	<5.7	<8.5	<4.8	<4.8	<4.8	<4.2	<4.7
Tilapia	07/863	<4	<4.6	12	<6.3	<22	<5.5	<3.7	<3.3	<3.9	<5.5	<5	<2.6

N.a. Not applicable
N.d. Not determined

Table II. 4 (continued). Levels of PBDEs in farmed fish samples (pg/g ww).

		BDE											
Sample and origin	IVM LIMS	138	153	154+ BB153*	156	183	184	191	196	197	209	Sum all	Sum EFSA-8
Salmon Scotland (UK) 1	07/785	<13	33	83	<17	<18	<14	<19	<26	<26	<24	1336	1083
Salmon Scotland (UK) 2	07/805	<18	24	69	<24	<26	<19	<27	<36	<36	<34	1235	1001
Salmon Norway 1	07/790	<16	31	79	<22	<23	<17	<25	<33	<33	59	1405	1165
Salmon Norway 2	07/804	<19	21	39	<25	<27	<20	<29	<38	<38	<36	1222	984
Salmon Norway 3	07/860	<34	<29	77	<31	<38	<43	<88	<66	<66	<70	1852	1520
Salmon Norway 4	07/870	<18	<16	35	<16	<20	<22	<46	<35	<35	45	626	526
Salmon Chili	07/810	<11	<9.2	<7.3	<14	<15	<11	<16	<21	<21	<20	75	75
Trout Italy	07/793	<4.3	3.9	7.3	<5.8	<6.1	<4.6	<6.5	<8.7	<8.7	22	413	393
Trout Denmark 1	07/807	<7.5	5.4	19	<7.5	<9	<9	<12	<11	<11	<19	321	270
Trout Denmark 2	07/861	<9.1	<7.9	21	<8.2	<10	<11	<23	<18	<18	<19	348	312
Trout Denmark 3	07/862	<11	<9.6	21	<10	<12	<14	<29	<21	<21	<23	396	338
Trout Turkey	07/869	<7.9	<6.8	12	<7.1	<8.8	<9.8	<20	<15	<15	3620	3831	3807
Shrimp Asia 1	07/789	2.7	1.8	<1.1	<2.1	<2.3	<1.7	<2.4	<3.2	<3.2	17	27	25
Shrimp Asia 2	07/794	<0.84	<0.73	<0.58	<1.1	<1.2	<0.88	<1.3	<1.7	<1.7	<1.6	N.a.	N.a.
Shrimp Asia 3	07/811	<1.4	<1.2	<0.98	<1.9	<2	<1.5	<2.1	<2.8	<2.8	14	19	19
Shrimp Bangladesh 1	07/797	<1.4	<1.3	<1	<1.9	<2	<1.5	<2.2	<2.9	<2.9	16	8	18
Shrimp Bangladesh 2	07/809	<0.68	<0.36	<0.29	<0.68	<0.81	<0.81	<1.1	<0.97	<0.97	7.8	9.7	9

		BDE											
Sample and origin	IVM LIMS	138	153	154+ BB153*	156	183	184	191	196	197	209	Sum all	Sum EFSA-8
Shrimp Netherlands	07/871	<2.5	1.8	2.5	<2.5	<3	<3	<4	<3.6	<3.6	16	61	59
Cod Norway	07/787	<1.9	<1.6	<1.5	<1.7	<2.1	<2.4	<4.9	<3.7	<3.7	<3.9	28	21
Pangasius Vietnam 1	07/786	<3.8	<3.3	<2.6	<5	<5.3	<4	<5.7	<7.5	<7.5	20	20	20
Pangasius Vietnam 2	07/791	<2.8	<2.4	<1.9	<3.7	<3.9	<3	<4.2	<5.6	<5.6	6.9	6.9	7
Pangasius Vietnam 3	07/796	<1.8	<1.5	<1.4	<1.6	<2	<2.2	<4.6	<3.4	<3.4	23	29	29
Pangasius Vietnam 4	07/806	<1.7	<0.93	<0.76	<1.7	<2.1	<2.1	<2.8	<2.5	<2.5	11	16	15
Pangasius Vietnam 5	07/864	<2.3	6.6	3.7	<2.3	6.4	<2.8	<3.7	<3.3	3.5	22	163	157
Pangasius Vietnam 6	07/868	<1.8	<0.98	<0.8	<1.8	<2.2	<2.2	<2.9	<2.6	<2.6	21	23	23
Pangasius Vietnam 7	08/002	<7.9	<4.2	<3.5	<7.9	<9.5	<9.5	<13	<11	<11	70	97	97
Tilapia Ecuador	07/788	<3.9	<3.4	<2.7	<5.2	<5.5	<4.1	<5.8	<7.8	<7.8	<7.4	N.a.	N.a.
Tilapia China 1	07/792	<2.8	<2.5	<2.9	<3.7	<4	<3	<4.2	<5.6	<5.6	<5.3	N.a.	N.a.
Tilapia China 2	07/808	<3.7	<3.3	5.0	<5	<5.3	<4	<5.6	<7.5	<7.5	<7.1	29	25
Tilapia Indonesia 1	07/795	<2.4	<1.3	<1.1	<2.4	<2.9	<2.9	<3.9	<3.5	<3.5	<6.2	6.5	6.5
Tilapia Indonesia 2	07/867	<2.6	2.1	2.1	<2.6	<3.1	<3.1	<4.1	<3.7	<3.7	<6.7	39	27
Tilapia Netherlands	08/001	<7.1	<3.8	3.2	<7.1	<8.5	<8.5	<11	<10	<10	<18	27	27
Tilapia	07/863	<4.9	<4.3	<4	<4.4	<5.6	<6.2	<13	<9.5	<9.5	<10	12	12

N.a. Not applicable

* Sum of BDE 154 and bromobiphenyl 153

Table II.5. Levels of α -, β- and γ-HBCD in farmed fish samples (ng/g ww).

IVM LIMS code	Sample and origin	α-HBCD	β-HBCD	γ-HBCD
07/785	Salmon Scotland (UK) 1	0.3	<0.1	<0.1
07/805	Salmon Scotland (UK) 2	0.2	<0.2	<0.2
07/790	Salmon Norway 1	0.3	<0.2	<0.2
07/804	Salmon Norway 2	0.2	<0.2	<0.2
07/860	Salmon Norway 3	0.3	<0.3	<0.3
07/870	Salmon Norway 4	0.2	<0.1	<0.1
07/810	Salmon Chile	<0.1	<0.2	<0.1
07/793	Trout Italy	0.3	0.05	0.01
07/807	Trout Denmark 1	0.05	<0.06	<0.07
07/861	Trout Denmark 2	0.07	<0.07	<0.07
07/862	Trout Denmark 3	0.1	<0.09	<0.09
07/869	Trout Turkey	0.1	<0.06	0.01
07/789	Shrimp Asia 1	<0.02	<0.02	<0.02
07/794	Shrimp Asia 2	0.006	<0.009	<0.009
07/811	Shrimp Asia 3	0.006	<0.02	<0.02
07/797	Shrimp Bangl 1	<0.02	<0.02	<0.02
07/809	Shrimp Bangl 2	<0.02	<0.01	<0.006
07/871	Shrimp Netherlands	0.7	0.5	0.003
07/787	Cod Norway	<0.01	<0.02	<0.02

IVM LIMS code	Sample and origin	α-HBCD	β-HBCD	γ-HBCD
07/786	Pangasius Vietnam 1	<0.02	<0.04	<0.04
07/791	Pangasius Vietnam 2	0.03	<0.03	<0.03
07/796	Pangasius Vietnam 3	<0.01	<0.02	<0.01
07/806	Pangasius Vietnam 4	<0.01	<0.02	<0.02
07/864	Pangasius Vietnam 5	0.01	<0.02	<0.02
07/868	Pangasius Vietnam 6	<0.009	<0.02	<0.02
08/002	Pangasius Vietnam 7	<0.04	<0.07	<0.07
07/788	Tilapia Ecuador	<0.02	<0.04	<0.04
07/792	Tilapia China 1	<0.02	<0.03	<0.03
07/808	Tilapia China 2	Nd	Nd	Nd
07/795	Tilapia Indonesia 1	<0.01	<0.02	<0.02
07/867	Tilapia Indonesia 2	<0.03	<0.07	<0.02
08/001	Tilapia Netherlands	<0.03	<0.06	<0.06
07/863	Tilapia	<0.02	<0.04	<0.04

N.d.: Not determined

Table II.6. Levels of PFCs in farmed fish samples (ng/g ww).

Sample and origin	IVM LIMS	PFHxA	PFHpA	PFOA	PFNA	PFDcA	PFUnA	PFDoA	PFTrA	PFTdA	PFBS	PFHxS	PFOS
Salmon Scotland (UK) 1	07/785	<0.7	<0.1	<0.7	<0.05	<0.10	<0.04	<0.09	<0.04	<0.5	<0.9	<1.0	<1.0
Salmon Scotland (UK) 2	07/805	<0.4	<0.08	<0.5	<0.04	<0.09	<0.05	<0.1	<0.05	<0.6	<0.7	<0.8	<0.8
Salmon Norway 1	07/790	<0.7	<0.1	<0.6	<0.05	<0.1	<0.07	<0.1	<0.07	<0.8	<1.0	<1.1	<1.1
Salmon Norway 2	07/804	<0.5	<0.09	<0.5	<0.04	<0.09	0.009	<0.08	0.04	<0.5	<0.6	<0.7	<0.7
Salmon Norway 3	07/860	<0.1	<0.03	<0.2	<0.1	<0.01	<0.01	<0.03	<0.03	<0.2	<0.2	<0.2	<0.2
Salmon Norway 4	07/870	<0.1	<0.03	<0.2	<0.2	<0.02	<0.04	<0.08	<0.08	<0.4	<0.3	<0.3	<0.3
Salmon Chile	07/810	<0.2	<0.03	<0.2	<0.2	<0.01	<0.01	<0.03	<0.03	<0.1	<0.2	<0.2	<0.2
Trout Denmark 1	07/807	<0.5	<0.10	<0.6	<0.04	<0.1	<0.05	<0.1	0.05	<0.7	<0.7	<0.8	<0.8
Trout Denmark 2	07/861	<0.2	<0.04	<0.2	<0.2	<0.02	<0.02	<0.03	0.04	<0.2	<0.2	<0.2	<0.2
Trout Denmark 3	07/862	<0.2	<0.03	<0.2	<0.2	<0.02	<0.02	<0.03	<0.03	<0.2	<0.2	<0.2	<0.2
Trout Italy	07/793	<0.9	<0.2	<0.9	<0.06	<0.1	<0.06	<0.1	<0.06	<0.7	<1.0	<1.2	<1.2
Trout Turkey	07/869	<0.1	<0.03	<0.2	<0.1	<0.01	<0.01	<0.02	0.07	<0.1	<0.2	<0.2	0.4
Cod Norway	07/787	<0.7	<0.1	<0.6	<0.05	<0.1	<0.1	<0.2	<0.1	<1.3	<1.2	<1.4	<1.4
Shrimp Asia 1	07/789	<0.4	<0.07	<0.4	0.01	0.02	0.1	0.03	0.3	0.07	<0.5	<0.6	<0.6
Shrimp Asia 2	07/794	<1.9	<0.3	<1.6	<0.1	<0.2	0.03	<0.2	0.05	<1.3	<1.8	<2.1	<2.1
Shrimp Asia 3	07/811	<0.3	<0.03	<0.1	<0.1	<0.01	0.08	<0.02	0.2	<0.1	<0.2	<0.2	<0.2
Shrimp Bangladesh 1	07/797	<1.7	<0.1	<0.8	<0.06	<0.1	<0.05	<0.1	<0.05	<0.6	<0.9	<1.0	<1.0

Sample and origin	IVM LIMS	PFHxA	PFHpA	PFOA	PFNA	PFDcA	PFUnA	PFDoA	PFTrA	PFTdA	PFBS	PFHxS	PFOS
Shrimp Bangladesh 2	07/809	<0.4	<0.04	<0.2	<0.2	<0.01	<0.01	<0.03	0.05	<0.2	<0.2	<0.3	<0.3
Shrimp Netherlands	07/871	<0.2	<0.03	<0.2	<0.2	0.03	0.1	0.07	0.08	<0.3	<0.2	<0.3	0.6
Pangasius Vietnam 1	07/786	<0.5	<0.08	<0.5	<0.04	<0.08	0.03	<0.08	0.08	<0.5	<0.6	<0.7	<0.7
Pangasius Vietnam 2	07/791	<0.4	<0.08	<0.4	<0.03	<0.07	0.02	<0.07	0.04	<0.4	<0.6	<0.6	<0.6
Pangasius Vietnam 3	07/796	<0.6	<0.09	<0.5	<0.03	<0.08	<0.04	<0.08	0.04	<0.5	<0.6	<0.7	<0.7
Pangasius Vietnam 4	07/806	<0.4	<0.06	<0.4	0.01	0.01	<0.03	<0.06	0.03	<0.4	0.1	<0.5	<0.5
Pangasius Vietnam 5	07/864	<0.1	<0.03	<0.1	<0.1	<0.01	<0.01	<0.02	<0.02	<0.1	<0.2	<0.2	<0.2
Pangasius Vietnam 6	07/868	<0.3	<0.03	<0.2	<0.2	<0.01	<0.01	<0.03	<0.03	<0.2	<0.2	<0.2	<0.2
Pangasius Vietnam 7	08/002	<0.1	<0.03	<0.2	<0.2	<0.02	<0.03	<0.07	<0.07	<0.4	<0.2	<0.3	<0.3
Tilapia Indonesia 1	07/795	<0.4	<0.08	<0.5	<0.03	<0.08	<0.04	<0.08	0.03	<0.5	<0.6	<0.7	<0.7
Tilapia Indonesia 2	07/867	<0.1	<0.03	<0.2	<0.2	<0.01	<0.01	<0.03	<0.03	<0.1	<0.2	<0.2	<0.2
Tilapia China 1	07/792	<0.5	<0.08	<0.4	0.02	0.03	0.05	<0.07	0.04	<0.4	<0.6	<0.7	<0.7
Tilapia China 2	07/808	<0.3	<0.03	<0.2	<0.2	<0.01	0.04	<0.02	0.08	<0.1	<0.2	<0.2	<0.2
Tilapia Ecuador	07/788	<0.5	<0.09	<0.5	<0.04	<0.1	<0.05	<0.1	<0.05	<0.6	<0.8	<1.0	0.2
Tilapia Netherlands	08/001	<0.2	<0.03	<0.2	<0.1	<0.01	0.02	<0.02	0.05	<0.1	<0.2	<0.2	<0.2
Tilapia	07/863	<0.1	<0.03	<0.1	<0.1	<0.01	<0.01	<0.02	0.05	<0.1	<0.2	<0.2	<0.2

N.d.: not determined

Table II.7. Levels of heavy metals in a selection of farmed fish samples (ng/g ww).

IVM-LIMS	Sample and origin	Cadmium	Mercury	Lead
07/793	Trout Italy	<2.7	8.1	<27
07/807	Trout Denmark 1	<2.4	34	<24
07/861	Trout Denmark 2	4.6	37	<23
07/862	Trout Denmark 3	<2.4	17	<24
07/869	Trout Tur	<2.9	70	<29
07/787	Cod Norway	<2.3	83	<23
07/786	Pangasius Vietnam 1	5.7	5.7	<19
07/791	Pangasius Vietnam 2	5.7	5.7	<19
07/796	Pangasius Vietnam 3	20	8	<20
07/806	Pangasius Vietnam 4	4.5	3	<15
07/864	Pangasius Vietnam 5	1.5	1.5	<15
07/868	Pangasius Vietnam 6	<1.4	7	<14
08/002	Pangasius Vietnam 7	<2.3	*2.3*	<23
07/788	Tilapia Ecuador	<2.7	5.4	<27
07/792	Tilapia China 1	13	2.1	<21
07/808	Tilapia China 2	<2.1	2.1	<21
07/795	Tilapia Indonesia 1	6.3	15	<21
07/867	Tilapia Indonesia 2	<2.1	17	<21
08/001	Tilapia Netherlands	14	27	<27
07/863	Tilapia	<2.1	17	<21

Table II.8. Results of microbiological screening of antibiotics in farmed fish samples. The positive samples were analysed by confirmation techniques and found negative.

IVM-LIMS	Sample and origin	VWA LIMS M08D00/	Sulfonamides	Tetracyclines	Quinolones	Macrolides	Beta-lactams
07/785	Salmon Scotland (UK) 1	1	-	-	-	-	-
07/805	Salmon Scotland (UK) 2	9	-	-	-	-	-
07/790	Salmon Norway 1	4	-	-	-	-	-
07/804	Salmon Norway 2	8	-	-	-	-	-
07/860	Salmon Norway 3	15	-	-	-	-	-
07/870	Salmon Norway 4	23	-	-	-	-	-
07/807	Trout Denmark 1	11	-	-	-	-	-
07/861	Trout Denmark 2	16	-	-	-	-	-
07/862	Trout Denmark 3	17	-	-	-	-	-
07/793	Trout Italy	5	-	-	-	-	-
07/869	Trout Turkey	22	-	-	-	-	-
07/787	Cod Norway	2	-	-	-	-	-
07/788	Tilapia Ecuador	3	-	-	-	-	-
07/808	Tilapia China 2	12	-	-	-	-	-
07/863	Tilapia	18	-	-	-	-	-
07/867	Tilapia Indonesia 2	20	-	-	-	-	-

IVM-LIMS	Sample and origin	VWA LIMS M08D00/	Sulfonamides	Tetracyclines	Quinolones	Macrolides	Beta-lactams
07/806	Pangasius Vietnam 4	10	+	-	-	+	-
07/864	Pangasius Vietnam 5	19	-	-	-	-	-
08/002	Pangasius Vietnam 7	25	+	-	-	-	-
07/868	Pangasius Vietnam 6	21	-	-	-	+	-
07/871	Shrimps Netherlands	24	+	-	-	-	-
07/794	Shrimps Asia 2	6	+	-	-	-	-
07/811	Shrimps Asia 3	14	-	-	-	-	-
07/797	Shrimps Bangladesh 1	7	-	-	-	-	-
07/809	Shrimps Bangladesh 2	13	+	-	-	-	-

Appendix III. Detailed information on contaminants levels in fish feed and feed ingredient samples

Table III.1. Levels of toxaphene congeners CHB 26, 50 and 62 in feed samples and feed ingredients (ng/g ww).

IVM-LIMS	VWA sampling code	Sample type	CHB 26	CHB 50	CHB 62	sum CHBs
08/356	66251969	Fish meal Norway	0.51	1.1	0.5	2.1
08/357	66251977	Fish meal Peru	0.03	0.04	<0.03	0.07
08/365	66251985	Fish oil	0.65	1.5	0.35	2.5
08/364	66251993	Palm oil	<0.03	<0.03	<0.19	<LOD
08/403	N.a.	Sunflower oil Ukraine	<0.03	<0.03	<0.19	<LOD
08/404	N.a.	Line seed oil	<0.03	<0.03	<0.2	<LOD
08/405	N.a.	Rape seed oil	<0.03	<0.03	<0.2	<LOD
08/366	66252094	Salmon feed	0.39	0.59	0.2	1.2
08/360	66252051	Trout feed (1)	0.2	0.43	0.19	0.8
08/361	66252078	Trout feed (2)	0.12	0.27	<0.09	0.39
08/367	66252108	Trout feed (3)	0.09	0.14	0.04	0.27
08/362	66252027	Tilapia feed (1)	0.03	0.06	<0.05	0.09
08/363	66252019	Tilapia feed (2)	0.32	0.61	0.24	1.17
08/370	66252132	Tilapia feed (3)	0.02	0.03	<0.03	0.05
08/373	N.a.	Shrimp feed	0.11	0.18	0.07	0.36

IVM-LIMS	VWA sampling code	Sample type	CHB 26	CHB 50	CHB 62	sum CHBs
08/358	66252035	Catfish feed (1)	0.28	0.56	0.22	1.06
08/359	66252043	Catfish feed (2)	0.06	0.13	<0.06	0.19
08/371	66252159	Catfish feed (3)	0.06	0.09	<0.03	0.15
08/372	66252167	Eel feed	0.11	0.18	0.05	0.34

N.a.: not applicable

Table III.2. Levels of mycotoxins feed samples, feed ingredients and shrimp samples (ng/g product).

IVM LIMS	Sample	Aflatoxin B_1	Deoxyniva-lenol	Fumonisin B_1	Fumonisin B_2	Fumonisin B_3	Zearalenone	Ergotamin	T2-toxin	HT2-toxin	Alternariol	Ochratoxi-ne-A
	Ingredients											
08/397	Corn (Brasil)	<0.5	<50	7200	1900	630	<10	<20	*<50*	*<50*	<10	<1
	Corn	<0.5	<50	1800	460	180	20	<20	*<50*	*<50*	<10	<1
	Corn gluten (1)	<0.5	2900	1400	300	110	180	<20	*<50*	*<50*	<10	<1
	Corn gluten (2)	0.90	<50	3600	1800	520	660	<20	*<50*	*<50*	<10	<1
	Wheat gluten (1)	<0.5	1100	<10	<10	<10	70	30	*<50*	*<50*	<10	<1
	Wheat gluten (2)	<0.5	2200	<10	<10	<10	110	<20	*<50*	*<50*	<10	<1
	Wheat (1)	<0.5	370	<10	<10	<10	25	<20	*<50*	*<50*	<10	<1
08/395	Wheat (2, UK)	<0.5	<50	<10	<10	<10	<10	1900	*<50*	*<50*	<10	6.5
08/399	Rapeseed (NL)	<0.5	<50	<10	<10	<10	<10	<20	*<50*	*<50*	<10	<1
08/400	Brassica (Australia)	<0.5	<50	<10	<10	<10	<10	<20	140	170	23	<1
08/398	Pea meal (EU)	<0.5	<50	<10	<10	<10	<10	<20	*<50*	*<50*	<10	<1
	Soy	<0.5	<50	<10	<10	<10	<10	<20	*<50*	*<50*	<10	<1
08/396	Soy beans (US/Canada)	<0.5	<50	<10	<10	<10	<10	<20	*<50*	*<50*	<10	<1
08/408	Soy bean meal (Argentina)	<0.5	<50	<10	<10	<10	54	<20	*<50*	*<50*	<10	<1
08/402	Palm oil (Indonesia)	<0.5	<50	<10	<10	<10	<10	<20	*<50*	*<50*	<10	<1
08/356	Fish meal (Norway)	<0.5	<50	<10	<10	<10	<10	<20	*<50*	*<50*	<10	<1
08/357	Fish meal (Peru)	<0.5	<50	<10	<10	<10	<10	<20	*<50*	*<50*	<10	<1

IVM LIMS	Sample	Aflatoxin B_1	Deoxynivalenol	Fumonisin B_1	Fumonisin B_2	Fumonisin B_3	Zearalenone	Ergotamin	T2-toxin	HT2-toxin	Alternariol	Ochratoxine-A
08/401	Fish meal	<0.5	90	<10	<10	<10	<10	<20	<50	<50	<10	<1
08/407	Fish oil	<0.5	<50	<10	<10	<10	<10	<20	<50	<50	<10	<1
	Fish feeds											
08/362	Tilapia (1)	<0.5	550	45	70	20	95	<20	<50	<50	<10	<1
08/363	Tilapia (2)	<0.5	260	<10	<10	<10	170	<20	<50	<50	<10	<1
08/370	Tilapia (3)	<0.5	100	<10	<10	<10	<10	<20	<50	<50	<10	<1
08/355	Catfish / Tilapia	<0.5	1500	800	210	95	80	<20	<50	<50	<10	<1
08/358	Catfish (1)	<0.5	250	<10	<10	<10	70	<20	<50	<50	<10	<1
08/359	Catfish (2)	<0.5	250	<10	<10	<10	40	<20	<50	<50	<10	<1
08/371	Catfish (3)	<0.5	140	<10	<10	<10	<10	<20	<50	<50	<10	<1
08/360	Trout (1)	<0.5	210	<10	<10	<10	85	<20	<50	<50	<10	<1
08/361	Trout (2)	<0.5	130	<10	<10	<10	<10	<20	<50	<50	<10	<1
08/367	Trout (3)	<0.5	<50	<10	<10	<10	15	<20	<50	<50	<10	<1
08/366	Salmon	<0.5	<50	<10	<10	<10	<10	<20	<50	<50	<10	<1
08/368	Common carp	<0.5	<50	20	20	<10	45	<20	<50	<50	<10	<1
08/369	Ide	<0.5	200	<10	<10	<10	<10	<20	<50	<50	<10	<1
08/372	Eel	<0.5	90	<10	<10	<10	20	<20	<50	<50	<10	<1
08/373	Shrimps	<0.5	60	<10	<10	<10	90	<20	<50	<50	<10	<1

IVM LIMS	Sample	Aflatoxin B_1	Deoxyniva-lenol	Fumonisin B_1	Fumonisin B_2	Fumonisin B_3	Zearalenone	Ergotamin	T2-toxin	HT2-toxin	Alternariol	Ochratoxi-ne-A
	Tissue samples											
07/789	Shrimps (1)	<0.5	<50	<10	<10	<10	<10	<20	<50	<50	<10	<1
07/794	Shrimps (2)	<0.5	<50	<10	<10	<10	<10	<20	<50	<50	<10	<1
07/811	Shrimps (3)	<0.5	<50	<10	<10	<10	<10	<20	<50	<50	<10	<1
07/797	Shrimps (4)	<0.5	<50	<10	<10	<10	<10	<20	<50	<50	<10	<1
07/809	Shrimps (5)	<0.5	<50	<10	<10	<10	<10	<20	<50	<50	<10	<1
07/871	Shrimps (6)	<0.5	<50	<10	<10	<10	<10	<20	<50	<50	<10	<1